UTAH DEPARTMENT OF ENVIRONMENTAL QUALITY

MERCURY SOURCE ASSESSMENT QUALITY PERFORMANCE PROJECT PLAN (QAPP)

April 2008

Preface:

This QAPP has been developed as a companion document to the Utah Department of Environmental Quality Mercury Source Protocol. The format of the QAPP is taken from "EPA Requirements for Quality Assurance Project Plans" (EPA QA/R-5. EOA/240/B-01/003, March 2001). There exists a newer 2005 version of the USEPA guidance document; however, it is form based and was determined to be a difficult format to use for this type of informational document. The SOPs have been incorporated into the document into appropriate sections below. Separate relevant SOPs can also be attached as they become available.

Table of Contents

A1. Problem Definition/Background

In August of 2005 Utah issued its first two fish consumption advisories for mercury in fish tissue (Gunlock Reservoir and Mill Creek). This was followed by a third advisory for mercury (Green River in Desolation Canyon) in November. In April of 2007, six additional fish consumption advisories were issued. Due to the high methylmercury levels found in the Great Salt Lake, waterfowl from the area were analyzed. As a result of high levels in three duck species, a health advisory for their consumption was issued. The mercury values in both fish and duck tissues are a result of mercury making its way into the waterbodies of Utah and moving up through the food chain. The key issues are the extent of mercury contamination in the state and source identification. The objective of this work is to efficiently characterize the mercury contamination and determine the likely source(s) of mercury to any waterbody in the State of Utah.

Background:

Mercury is a naturally occurring metallic element that is found in soil, air, and water. It is present in many forms such as elemental or metallic mercury, inorganic mercury compounds, and organic mercury compounds. Mercury combines with elements, such as chlorine, sulfur, or oxygen to form inorganic mercury compounds or it may combine with alkyl and aryl organic groups to form organic mercury compounds. The most common and prevalent form of organic mercury in the environment is methylmercury. It is produced by microscopic organisms that convert inorganic mercury in the soil and water into methylmercury. Thus, the more mercury that is present or released into the environment, the more chance that methylmercury is produced by these organisms. Methylmercury is important because it bioaccumulates in the food chain. This is demonstrated by the tissue levels observed in some fish and waterfowl in the State of Utah.

The mercury released (from sources) to the environment is most likely inorganic, some of which will convert to organic methylmercury through microbial activity. When higher levels of inorganic mercury are available, higher levels of methylmercury are expected to be formed. Methylmercury is easily taken up by aquatic plants and animals. As larger organisms continue to feed on these smaller organisms, methylmercury is concentrated up the food chain in a process called bioaccumulation. It is bioaccumulation which drives this study. This Quality Assurance Project Plan will provide uniform methodologies for sampling and analyses of various media throughout the State of Utah to provide comparable, defensible data.

Efforts to characterize the extent of mercury contamination have been ongoing in Utah. Over 1,100 fish samples from 220 bodies of water have been analyzed. (Whitehead, Mercury WorkGroup Presentation, January 2007). Eleven percent of these tissue samples had mercury levels exceeding 0.3mg/kg. The Utah Department of Health has adopted the EPA screening value for mercury in fish of 0.30 mg/kg as the level of concern or action level for methylmercury concentration in fish tissue. (EPA Fact Sheet 2001)(USEPA Fish Advisories)

A2. Project Task/Description

The specific objectives of this study are to provide standardized guidelines to be followed in the investigation of the source(s) of mercury contamination in Utah. For each media type the following are provided:

1. Detailed sampling plans

2. Detailed sample handling requirements from field to laboratory

3. Analytical methods

A3. Quality Objectives and Criteria

Data Quality Objectives are quoted from the State of Utah Quality Assurance Program Plan, Revision 1, November 2006 for the Division of Solid and Hazardous Waste (DEQ, 2006).

Data Completeness

Completeness is defined as the amount of valid data obtained from a measurement system compared to the amount that is expected to be obtained. A goal of at least *95%* completeness should be obtained.

Data Accuracy

Accuracy is the degree of agreement between a measurement and an accepted reference or true value. The accuracy is determined from analyses of samples spiked with a known concentration. The number of spiked samples and the spiking levels will be taken from the respective methods.

The formula used to assess the accuracy of a laboratory control spike (LCS) is:

$$\%R = (Q_{LCS} / Q_{KC}) \times 100$$

Where: %R = Percent Recovery
Q_{LCS} = Quantity of Analyte Found in the Spike Sample
Q_{KC} = Known Concentration of the LCS

The formula used to assess the accuracy of the matrix spike/matrix spike duplicates (MS/MSD) samples is:

$$\%R = ((Qss - Qus)/Qs) \times 100$$

Where: %R = Percent Recovery
Qss = Quantity of Analyte Found in the Spike Sample
Qus= Quantity of Analyte Found in the Unspiked Sample
Qs = Quantity of Added Spike

Calculation of the accuracy for each analysis will be based on different criteria as discussed in the Quality Assurance Project Plan and the analytical methods. The default value for water is ± 25% and for soil/solid is ± 40%, project specific requirements may vary from the default values due to other considerations.

Data Precision

Precision is defined as the degree of mutual agreement among individual measurements made under prescribed conditions. Precision will use two different measurements depending on the number of data points being considered. Two data points will have the relative percent difference (RPD) calculated. Three or more data points will use the relative standard deviation (RSD) as a measure of the precision.

$$RSD=100s/x_{avg}$$

Where: RSD = Relative Standard Deviation
s=Standard Deviation
x_{avg}= Average Result of Sample

External precision audits may be conducted by submitting blind duplicates to the laboratory and comparing the results with the acceptance criteria. The number of blind duplicates required will usually be 20 percent of all samples taken. Precision will be calculated for laboratory or field samples using the following equations:

$$RPD= \{(X_1-X_2)/[(X_1+X2)/_2]\} \times 100$$

Where: RPD = Relative Percent Difference
X_1 = Highest Analytical Result of Sample
X_2 = Lowest Analytical Result of Sample

Calculation of the precision for each analysis will be based on different criteria as discussed in the project plan and the analytical methods. The default value for water is <20% and for soil/solids is <40%; project specific requirements may vary due to other considerations.

Data Representativeness

To assure representativeness, all samples must be taken following protocols as set forth in Standard Operating Procedures for Field Samplers, Samplers and Sampling Procedures for Hazardous Waste Streams (USEPA), this QAPP, or other procedures approved by the Project Manager. Also, site descriptions, site photo documentation, and sampling conditions and techniques may be documented in bound field notebooks.

Data Comparability

Comparability is a quantitative characteristic, which may be considered in planning sampling activities. The project manager will work closely with the State Health Laboratory to ensure all data generated are consistent with and expressed in the same units as the data generated by other laboratories reporting similar analyses. Soils, solids, and sludges must be reported on a dry weight basis. Biological tissue (e.g. fish or duck tissue) must be reported on a wet weight basis. This will allow comparison of data among organizations.

Similarly, the project manager will work closely with the field team to ensure that all data generated by field measurements are expressed in units that are consistent with standard practices. In addition to units, comparability will be assured in terms of sampling plans, analytical methodology, quality control and data reporting. The Data Quality Objectives (DQOs) found in Table 1 are consistent with the State of Utah DEQ Quality Assurance Program Plan.

Table 1. Data Quality Objectives

from the State of Utah DEQ Quality Assurance Program Plan

Sample Media	Parameter	Method	Reporting Limit	Accuracy	Precision	Completeness
Wet Air Deposition	Hg (Total)	USEPA 1631e	Based on DL	75% - 125%	+/- 42% RPD	95%
Dry Air Deposition	RGM, Hg^0	7473	1.0ng/m^3	60% - 140%	+/- 40% RPD	95%
Particulate in Air	Hg_P	7473	1.0ng/m^3	60% - 140%	+/- 40% RPD	95%
Surface Water	Hg (Total)	USEPA 1631e	0.5 ng/L	75% - 125%	+/- 20% RPD	95%
Surface Water	Hg (Dissolved)	USEPA 1631e	0.5 ng/L	75% - 125%	+/- 20% RPD	95%
Geothermal Water	Hg (Total)	USEPA 1631e	0.5 ng/L	75% - 125%	+/- 20% RPD	95%
Snowpack	Hg (Total)	USEPA 1631e	0.5 ng/L	75% - 125%	+/- 20% RPD	95%
Soil	Hg (Total)	7471	0.04mg/Kg (dry)	60% - 140%	+/- 40% RPD	95%
Sediment	Hg (Total)	7471	0.04mg/Kg (dry)	60% - 140%	+/- 40% RPD	95%
Soil	Hg (Total-HgS)	245.5	0.04mg/Kg(dry)	60% - 140%	+/- 40% RPD	95%
Sediment	Hg (Total-HgS)	245.5	0.04mg/Kg(dry)	60% - 140%	+/- 40% RPD	95%
Fish Tissue	Hg (Total)	7473	0.1µg/Kg (wet)*	60% - 140%	+/- 40% RPD	95%
Invertebrate Tissue	Hg (Total)	7473	0.1µg/Kg (wet)*	60% - 140%	+/- 40% RPD	95%
Moss	Hg (Total)	7473	0.1µg/Kg (wet)*	60% - 140%	+/- 40% RPD	95%
			*Assuming a 0.2g sample size			

Quality assurance (QA) criteria for this project are discussed throughout this document. Some of the major QA criteria for this project are seen in Table 2:

Table 2. Data Quality Assurance Criteria

Sample Media	**Parameter**	**Method**	**Reporting Limit**	**Equipment Blank**	**Field Blank**	**Field Duplicate**	**Matrix Spike**
Wet Air Deposition	Hg (Total)	USEPA 1631e	Based on det'd DL	1/sampling event	1/quarter	1/20	1/10
Dry Air Deposition	RGM, Hg^0	7473	1.0ng/m^3	NA	1/10	1/10	NA
Particulate in Air	Hg_p	7473	1.0ng/m^3	NA	1/10	1/10	NA
Surface Water	Hg (Total)	USEPA 1631e	0.5ng/L	1/sampling event	1/10	1/10	1/10
Surface Water	Hg (Dissolved)	USEPA 1631e	0.5ng/L	1/sampling event	1/10	1/10	1/10
Geothermal Water	Hg (Total)	USEPA 1631e	0.5ng/L	1/sampling event	1/10	1/10	1/10
Snowpack	Hg (Total)	USEPA 1631e	0.5ng/L	1/sampling event	1/10	1/10	1/10
Soil	Hg (Total)	7471, 245.5	0.04mg/Kg	NA	NA	1/20	1/20
Sediment	Hg (Total)	7471, 245.5	0.04mg/Kg	NA	NA	1/20	1/20
Fish Tissue	Hg (Total)	7473	0.1µg/L*(wet)	1/sampling event	1/10	1/10	1/10
Invertebrate Tissue	Hg (Total)	7473	0.1µg/L*(wet)	1/sampling event	1/10	1/10	1/10
Moss	Hg (Total)	7473	0.1µg/L*(wet)	NA	1/10	1/10	1/10
			*Assuming a 0.2g sample size				

A4. Special Training and Certification

If speciation of mercury in air is desired, special training is required with the Tekran Model 1135 instrument.

A5. Documentation and Records

Sample Receipt and Log-in Procedures
Separate field data sheets will be maintained for each sampling event. Samples must be accompanied by a form with the following information: sampling location, lat/long, sampling point

description, date sampled, time sampled, sampler, weather condition, container number, equipment ID numbers and analyses requested.

Upon receipt, the laboratory verifies the information contained on the Request for Analysis form and checks to make certain that samples meet appropriate handling and preservation requirements by:

- Matching actual sample container numbers with those listed on *Request for Analysis* form;
- Checking that appropriate containers were used for the analytes requested;
- Testing pH to determine whether samples requiring acid were preserved correctly;
- Verifying condition of samples as received.

Samples improperly documented, preserved, or exceeding holding time are either rejected by the laboratory for analysis, or analyzed and the result reported as an "*estimate*." The sampler is notified and re-sampling is recommended.

Chain-of-Custody or Sample Tracking

Samples may use either a legal chain-of-custody or sample tracking form to enable tracking the possession and handling of a sample during transfer, (from sample collection through laboratory analysis and final disposal), so that their physical possession is known at all steps in the process. Refer to the current version of EPA's Guide for Field Samplers for updated tracking information.

A sample is under legal chain-of-custody if:

I. It is in the person's possession, or
2. It is in the person's view at all times, or
3. It is locked in a secure location.

Sample Tracking Procedures:

1. Sample identification (e.g., Division sample number)
2. Sample description (e.g., location and depth, if applicable)
3. Sample date and time
4. Sample matrix (e.g. air, water etc.)
5. Sampler and Division employee if not sampler
6. Analytes requested methods, and special instructions if needed
7. Contact information

At the laboratory, samples are logged in and identified as either legal chain-of-custody or sample tracking samples. The laboratory will follow the sample handling procedure appropriate to the sample, e.g., chain-of-custody procedures. Samples will remain in the control of the sampling crew until delivery to the laboratory.

Field Notebook

A bound field notebook will be maintained by the sampling teams to provide a daily record of significant events, observations, and measurements during field investigations. This record will include water level data, field measurements, personnel, weather observations, and physical conditions of the sampling environment and site. All entries in the field notebooks must be signed in waterproof ink and dated. The field notebooks will be kept as a permanent record.

Corrections to Documentation

All original data recorded in field notebooks and other forms will be written in waterproof ink. None of these documents will be destroyed or thrown away, even if they are illegible or contain inaccuracies that require a replacement document. If an error is made on a document assigned to one individual, that individual will make corrections by crossing a single line through the error, entering the correct information, and initialing and dating the correction.

DATA GENERATION AND ACQUISITION

B1. Sampling Process Design

The USEPA Data Quality Objectives Process (USEPA, 2000) will be used to determine the most efficient sampling design. Several types of sampling designs may be used including Authoritative, Simple Random, Stratified Random, and Systematic Random. Random sampling will be the preferred methodology because inferential statistics are based on random samples. However, in some instances the sampling may not be random (e.g., selecting the largest fish when electrofishing). In these cases, the potential for bias will be discussed. No bias is desirable but a low bias for mercury concentrations has higher consequences than a high bias because of the potential to underestimate human health risks.

The screening level for mercury in fish is 0.3 mg/kg. The null hypothesis is that the average mercury concentration in fish is less than or equal to 0.3 mg/kg. The false positive error rate was set to a maximum of 0.1, i.e., if we conclude that mercury concentrations are greater than 0.3 mg/kg, there is a maximum of a 10 percent chance that the mean concentration is really less than or equal to 0.3 mg/kg. The false negative probability was set to 0.2, i.e., if the mean mercury concentration is greater than the minimum detectable difference (see next paragraph), there is an 80 percent chance that we would correctly conclude that the mean concentration is greater 0.3 mg/kg.

A minimum detectable difference must be selected (USEPA, 2000). The concentrations between the screening level and minimum detectable difference are called the gray region. Concentrations in the gray region are “too close to call” and relatively high rates of decision errors are acceptable. The minimum detectable difference was set to 0.15 mg/kg, i.e., the gray region is from 0.3 to 0.45 mg/kg.

The number of fish samples will be considered adequate when these minimum criteria are met. Note that these are minimum criteria. When a greater number of samples are collected, the variance is lower, or the mercury concentrations are greater than 0.45 mg/kg, the probability of decisions error decrease significantly.

B2. Sampling Methods

Sampling will be accomplished using standard USEPA methods when available. When standard methods are not available, A detailed explanation of the sampling will be documented.

Specific sample preservation methods and holding times are summarized in Table 3.

Table 3. Sample Preservation Methods and Holding Times

Medium	Sampling Reference	Number of Samples	Holding Time	Container	Sample Preservation
Wet Air Deposition	USGS 2004	Dependent on availability of sampling units	90 days	0.5 Liter (glass or fluoropolymer)	4°C; 5 ml/L BrCl(0.2%) within 48 hours
Dry Air Deposition	Munthe 1996	Dependent on availability of sampling units	28 days	Sealed in Glass Jars	Silver Wool in Jar
Particulate in Air (Hg_p)	Munthe 1996	Dependent on availability of sampling units	28 days	Petri Dish sealed with Teflon Tape	Frozen
Surface Water (Total)	USEPA 1669	8 Samples per location	90 days	0.5 Liter (glass or fluoropolymer)	4°C; 5 ml/L BrCl(0.2%) within 48 hours
Surface Water (Dissolved)	USEPA 1669	8 Samples per location	90days	0.5 Liter (glass or fluoropolymer)	0.45 Filter prior to preservation; 4°C; 5 ml/L BrCl(0.2%) within 48 hours

Medium	Sampling Reference	Number of Samples	Holding Time	Container	Sample Preservation
Geothermal Water	USEPA 1669	8 Samples per location	90days	0.5 Liter (glass or fluoropolymer)	4°C; 5 ml/L BrCl(0.2%) within 48 hours
Snowpack	QAPP Section B2.8	8 Samples per location	90 days	4x 1-gal Ziploc bags; then clean 0.5L Glass	4°C; 5 ml/L BrCl(0.2%) within 48 hours
Soil	QAPP Section B2.9	8 Samples per location	28 days	250 ml Glass	Chill, 4°C
Sediment	QAPP Section B2.10	8 Samples per location	28 days	250 ml Glass	Chill, 4°C
Fish	Utah DEQ SOP & QAPP B2.11	8 Samples per location	90 days	200g Ziploc Bags	Frozen
Invertebrates	QAPP Section B2.12	8 Samples per location	90 days	200g Ziploc Bags	Frozen
Moss	QAPP Section B2.13	8 Samples per location	90 days	5.0g Ziploc Bags	Frozen

B2.1 Sampling of Mercury in Precipitation and Air

Introduction

Sampling for mercury in the atmosphere requires the sampling of both precipitation and air. Precipitation samples allow for estimates of wet deposition of Particulate Phase Mercury (Hg_p), Reactive Gaseous Mercury (RGM), and Elemental Mercury (Hg^0) while air samples are used in developing concentrations and estimated dry deposition flux for these species. Both types of sampling must be performed in tandem at the same monitoring station to support the calculation of total mercury loads over the sampling period.

In many cases, proven techniques and methods exist that are directly applicable to the Utah mercury sampling protocol. Still, researchers are working to improve the sampling of mercury particulates in air and to reduce the costs associated with the speciation of mercury compounds in the atmosphere. Also, because dry deposition is known to be an important component of total mercury deposition, other research efforts are focused on its direct measurement.

The risk of sample contamination is high when working with such low levels of mercury. Care must be taken to minimize the potential for contamination, which could compromise the quality and the usability of the samples leading to increased sampling costs. This underscores the need to fully integrate the clean hands/dirty hands protocol (USGS, 2004).

Additional references provide knowledge, insight, and guidance about sampling and analyzing mercury in air. Some of these include: Bieber and Althoff (1995), Fitzgerald and Gill (1979), Iverfeldt (1991a, 1991b, 1993, 1992, 1994), Landis, Stevens, Schaedlich, and Prestbo (2002), USEPA (1999), and Winkler (1997).

Sampling Methods for Mercury in Precipitation

The guidelines for the sampling of mercury in precipitation provided by Munthe (Munthe, 1996) and (OSPAR, 1997) will serve as the basis for this effort. As in all types of sampling performed as part of the Utah mercury sampling protocol, the clean hands/dirty hands protocol (USGS, 2004 TWRI Book 9-A5, NFM 4.0.1) is incorporated by reference. Further, sampling must be conducted in accordance with Section 4.0, Sampling Procedures, of the Division of Solid and Hazardous Waste Quality Assurance Program, Revision 1 (DSHW QAP) (Utah DEQ, 2006).

Sampler Design and Materials

Mercury is collected in special precipitation samplers. Two alternative materials may be used for funnels and collection bottles: borosilicate glass and a halocarbon such as Teflon®. Borosilicate glass is preferred due to lower costs and general availability. Quartz glass may be used but is generally avoided due to its higher cost.

Precipitation can be sampled using either wide-mouthed jars or funnels and bottles; the use of a funnel/bottle combination with a capillary tube between the funnel and bottle is preferred. The funnel/bottle combination minimizes the potential for contamination by limiting Hg^0 diffusion from the atmosphere and preventing dust, birds, insects, etc. from entering the sample during and after collection. Funnel area and bottle sizes should be modified to suit the sampling period used.

The collection vessels must be shielded from light to avoid photo-induced reduction of mercury on the walls of the sample bottle. Unless filtered versus dissolved mercury speciation is desired, the sample bottles must contain preservative. If speciation is desired, the preservative is added once filtration is completed in the field.

For drier climates where precipitation events are unpredictable, the preferred collector is a wet-only automated system that opens when precipitation begins and closes when it ends. Such samplers are thermally controlled to deal with changing conditions throughout the year. Wet-only samplers are used by the German national monitoring program as well as by research groups working in the Great Lakes area (Landis and Keeler, 1996) and in the US National Atmospheric Deposition Program (NADP) (Vermette et al., 1995). Wet-only samplers have the advantage of avoiding particle dry deposition. Because sample collection must be correlated with parallel measurements of precipitation, a standard rain gauge must be included with the sampling equipment. The wet-only sampling equipment currently recommended by the US National Atmospheric Deposition Program (NADP) with requirements discussed by Vermette (Vermette et al., 1995) is recommended for the Utah mercury sampling protocol.

Washing Procedure for Glass Equipment

All glass equipment used for sampling or for storing water samples for mercury analysis must be cleaned extensively before use. The glassware is washed thoroughly according to the procedure outlined in Table 4.

Plastic gloves must be used during all steps of the washing procedure. Glassware that is not being leached in tanks must always be stored in double plastic bags.

Table 4. Washing Procedure for Glass Equipment When Sampling Mercury in Precipitation

Step	Procedure
1	Leach in an alkaline detergent for 24 hours. Rinse thoroughly with de-ionized water
2	Leach in a solution of HNO_3 (Puriss) and de-ionized water (1:3) for 7 days. This must be done in a polyethylene tank placed under a fume hood. Rinse thoroughly with de-ionized water.
3	Leach in a 0.1 M solution of HNO_3 (P.A.) in high-purity water (e.g. Milli-Q) for 7 days. Rinse thoroughly with de-ionized water.
4	Fill bottles with high purity water. Add 5 ml/L BrCl (0.2%) solution. Let stand for at least 24 hours.
5	Add 5 ml/L 12% $NH_2OH \cdot HCl$. Let stand for 1 hour.
6	Pour out the above solution. Rinse with large amounts of de-ionized water followed by large amounts of high-purity water.
7	Add 2.5 ml 30% HCl (Suprapur) to the bulk sampler bottles (0.5 L borosilicate glass) All other bottles and containers must be filled with 5 ml/L 30% HCl (Suprapur). Other glassware must be air-dried in a clean zone.

Bottles that are believed to be contaminated are baked at 500^{o}C for 5 hours and then washed according to the procedure above, excluding Step 1.

Sampling Procedure

All samples and replacement sample bottles must be handled with care in order to avoid contamination during transport and storage. Sample bottles must only be handled using plastic gloves and all bottles will be kept in double plastic bags during transport and storage.

Before sending the precipitation bottle to the field, it will be filled with 5 ml/L BrCl (0.2%). Take care when handling these bottles to avoid contamination.

The sampling procedure for mercury in precipitation is outlined in Table 5 and summarized in Table 6. This procedure is recommended for the funnel/bottle samplers described above. For alternative sampling devices, this procedure can be adapted.

Table 5. Sampling Procedure for Mercury in Precipitation

Step	Procedure
1	Bring a new collection bottle, plastic bags and container (squeeze bottle) with high purity water to the sampler. All equipment needed for the bottle exchange will be placed on a plastic cover either on the ground or on an available surface.
2	Remove outer plastic tube.
3	Open double bags of the new collection bottle (Leave the bottle in the bags).
4	Carefully remove the ground glass joint connecting the bottle to the capillary tube. Use both hands, one for loosening the glass fitting the other for holding the funnel.
5	Remove the stopper from the new collection bottle and close the bottle containing the precipitation sample. This bottle is then put in double plastic bags.
6	Without mounting the new bottle, rinse the funnel and capillary with high purity water. If visible materials (dust, insect etc.) are present, disconnect the funnel from the capillary and rinse separately. New plastic gloves must be used if handling the watch glass or touching the inside of the funnel is necessary. If the funnel and capillary are visibly dirty even after rinsing, they must be exchanged with newly washed pieces.
7	Remove the new collection bottle from the plastic bags and place it in the plastic casing. Connect the ground glass fitting and check all connections. Make sure that all connections are without gaps so that the silicon tubing is not unnecessarily exposed to the precipitation sample.
8	Replace outer plastic tube.

Samples for storage must be stored in double zip-lock plastic bags, refrigerated, kept in the dark, and transported to the laboratory. Sample holding time is 90 days when preserved in this manner.

Quality Control – Quality Assurance

All sampling activities must be conducted in accordance with the QAPP. In addition, written instructions will be provided to personnel on how to avoid contaminating the samples. Replacement parts must be easily available so that glassware can be exchanged if contamination is suspected.

The risks of contamination when using bulk samplers and extended sampling times are controlled by using two or three samplers in parallel. Contaminated samples can then be identified and discarded and the corresponding data excluded from consideration.

Field Blanks

Field blanks will be taken at least four times each year.

Two extra sampling bottles must be brought to the site; one containing dilute HCl (pH 3 to 4) and one empty. After removing the regular sample bottle the empty bottle will be installed and the dilute HCl poured through the sampling device (*e.g.* funnel and capillary). The bottle will be stoppered, double bagged and brought to the laboratory for analysis. The mercury content of the dilute HCl will be compared to that of samples stored in a clean laboratory environment. If the blank values exceed 20% of the concentrations normally measured at the site, steps must be taken to reduce the blank concentrations (e.g., exchanging or cleaning the sampling devices); resampling or qualification of the data as 'estimates' will be required.

The yearly average blank value is used to determine the detection limit and will be reported to appropriate project personnel.

Special Problems

1. When first starting to sample precipitation for mercury analysis numerous problems can arise, mostly associated with high blanks. Weekly sampling will be undertaken initially, even if monthly sampling is planned. In this way the number of sampling periods without results can be reduced.

2. The most common causes of sample contamination are insects, bird droppings or other material in the sampling vessels. This is the major drawback to bulk sampling. In areas with large numbers of birds in the vicinity, it may be necessary to install devices that prevent birds from perching atop the samplers.

3. When using extended sampling periods, the risks of contamination can be controlled by employing two or three samplers in parallel. Contaminated samples can then be identified and the results discarded, minimizing the loss of information. Contamination of two samplers in parallel is very rare.

4. When temperatures are high, it may be necessary to cool the sample bottle to prevent the evaporation of mercury from the sample.

Table 6. Precipitation Sampling Summary

Item	Recommendation	Acceptable Alternative
Material	Borosilicate glass.	Halocarbon materials, quartz.
Sampler Design	Bulk or wet-only samplers with gaseous Hg prevention and light shield. Heating and/or cooling of sample bottle depending on climatic conditions.	Event sampling using funnels and bottles or jars.
Sampling Time	1 week to 1 month.	N/A
Preservation of Samples	Monthly sampling 5 ml/L HCl (Suprapur) prior to sampling.	In sampling periods of <2 weeks and samples are cooled if necessary.
QA/QC	Field blanks. Written instructions for field personnel. Sample in accordance with the DSHW QAP.	N/A

B2.2 Sampling Methods for Total Gaseous Mercury in Air

For all types of sampling performed as part of the Utah mercury sampling protocol, the clean hands/dirty hands protocol (USGS, 2004 TWRI Book 9-A5, NFM 4.0.1) is incorporated by reference. Further, sampling must be conducted in accordance with Section 4.0, Sampling Procedures, of the QAPP (Utah DEQ, 2006).

Sampler Design and Cleaning Procedure

The sampling of total gaseous mercury (RGM plus Hg^0) in air is usually done using gold traps with gas meters or mass flow controllers for air volume measurements. During the last few years, automated instruments for the sampling and analysis of mercury in air have been made available. The automated method applies the same basic principles as the manual method and has been shown to generate comparable results (Schroeder et al., 1995; Ebinghaus et al., 1999).

Gold traps are comprised of 10 to12 centimeter quartz glass tubes filled with gold adsorbent. The gold adsorbent can be small pieces (1-2 mm) of 1 mm solid gold wire mixed with a crushed-quartz glass bearer or, alternatively, sand, glass beads or quartz glass coated with a thin layer of gold. The latter trap type usually generates lower blank values.

The gaseous mercury collected with the gold trap in the field is transferred by heating to a calibrated gold trap, using Hg-free argon with a purity greater than 99.998% (at a flow rate of 30 ml/min) as the carrier gas. This is known as the dual amalgamation technique. The sampling and transfer lines are made of Teflon® tubing. Glass to glass connections (i.e. between the sampling trap and the analytical trap) are made with silicone tubing. The inlet filter is a quartz tube 75 mm in length (with an inner diameter of 4 mm and an outer diameter of 6 mm) with a quartz wool plug 15 mm in length. A brief summary of the equipment is as follows:

- Preferred materials are glass, quartz, and Teflon®;
- A heating device (e.g., Perkin Elmer Part No. 102961 or equivalent) with a variable transformer will be used;
- Argon gas with purity greater than 99.998 % by volume; and
- An Automatic Dräger gas detector pump with one stroke volume = 100 ± 5 cm^3.

Cleaning procedures are outlined in Table 7.

Table 7. Cleaning Procedures for Equipment Used in Sampling Total Gaseous Mercury in Air

Item	Procedure
Field Trap	Thermal desorption at 500ºC for three minutes while purging with a Hg-free argon stream at a flow rate of 30 ml/min (three consecutive times)
Analytical Trap	Thermal desorption at 800ºC for 25 seconds while purging with a Hg-free argon stream at a flow rate of 30 ml/min (two consecutive times)
Inlet Filter	Thermal desorption at 1200ºC for 1.5 minutes while purging with an air-stream of 30 ml/min (three consecutive times)

Sampling Procedure

The site must be chosen with great care to avoid contamination or non-representative results. Sampling must be performed at a minimum of 1.5 meters above the ground or nearby structure (e.g., wall) in order to avoid the influence of local fluxes. The sampling system contains one protective quartz wool plug followed by two gold traps in series.

The analytical method for sampling total gaseous mercury (including elemental, organic and inorganic mercury) is based on the amalgamation of mercury with gold. Total gaseous mercury is collected on the surface of the gold. For sample collection, two of these traps are placed in series. With this arrangement a breakthrough of mercury is detected when a significant amount of mercury is found on the second trap.

The sampling time and air volumes must be sufficient to collect enough mercury for analysis but not so large as to cause a breakthrough of mercury. Sampling flow rates in the range 0.1 to 0.5 L/min and maximum volumes of 100 to 1000 liters are normally adequate.

The general configuration for the set-up of the sampling system for total gaseous mercury in air is:

- One quartz wool plug followed by two field traps in series;
- Sample flow rate of 0.1 to 0.5 ml/min; and
- Sampling time between 12 and 24 hours [for TGM concentrations (RGM + Hg^0) in the range of 1 to 10 ng/m^3].

Sample Storage

Exchange field traps according to a fixed procedure taking great care to avoid contamination. Before and after sampling, close the ends of the traps with plastic caps and store the traps in a firmly closed glass bottle. To prevent contamination during storage, one gram of silver wool must be kept in the bottle to bind gaseous mercury diffusing into the storage vessel.

Volume Standardization

Use the Ideal Gas Law to convert the sample volume to a volume at standard conditions. For example, at 0 ºC and 1 atmosphere (atm) [equivalent to 273.15ºK and 1013.25 millibar (mB)], the conversion is made using the following equation:

$$V_{Std} = V_{current} \times (273.15\ ºK / T_{inlet}) \times (P_{inlet} / 1013.25\ mB).$$

Quality Control - Quality Assurance

All sampling activities must be conducted in accordance with the DSHW QAP.

Additional quality control steps are primarily associated with gold trap collection and analytical instrument reliability. All gold traps must be individually calibrated at regular intervals. This is most conveniently done using a source of gaseous mercury (e.g., a thermostated vessel containing liquid mercury) from which gaseous samples can be drawn with a gas tight syringe. Gold traps with low recovery must be discarded.

Alternative methods for sampling mercury in air are not generally available. However, commercially available iodated carbon traps have been successfully used for sampling over extended periods (i.e*.,* days) (Bloom et al*.,* 1995).

Table 8 summarizes the sampling procedure for gaseous mercury.

Special Problems

1. Under certain conditions, breakthrough of mercury can occur at air volumes considerably smaller than those recommended above. This is usually due to the presence of trace constituents in the air which block the gold surface. Possible contaminants are sulphur-containing volatile organic compounds and volatile inorganic species capable of forming solid salts on the gold surface via atmospheric reactions (*e.g.* $(NH_4)_2SO_4$). If this happens, considerably smaller sampling volumes (e.g., less than 100 liters) must be used.

2. When using automated systems, frequent re-calibration is necessary and this frequency will vary according to the temporal resolution of the sampling. Daily re-calibration is a minimum.

Table 8. Gaseous Mercury Sampling Summary

Item	Recommendation	Acceptable Alternative
Material	Solid gold traps.	Coated gold traps.
Sampling Design	1 protective quartz wool plug followed by 2 gold traps in series.	Teflon filter or quartz fiber filter followed by 2 gold traps in series.
Air Flow Rate	100 to 500 ml/min.	N/A
Sample Volume	100 to 1000 liters.	N/A
QA/QC	2 gold traps in series to check mercury breakthrough. Sample in accordance with the DSHW QAP.	N/A

B2.3 Sampling Methods for Particulate Phase Mercury in Air

Sampling of particulate phase mercury in air is one of the difficult steps in measurements of atmospheric mercury. This is due to errors that may occur during both sampling and analysis. In ambient air the particulate fraction of mercury usually is less than 5%, with volatile mercury making up the reminder. This increases the risk of gas to particle conversion during sampling. For this reason, sampling of particulate phase mercury must be considered as an operationally defined method. The presence of particulate mercury in air will greatly influence the overall atmospheric deposition of mercury and the ongoing development of these techniques is important in furthering the understanding of these important processes. The sampling procedure developed by Keeler and Landis (Keeler, 1994 and USEPA, 1999) is used as the basis for the sampling of particulate mercury in air. As in all types of sampling performed as part of the Utah mercury sampling protocol, the clean hands/dirty hands protocol (USGS, 2004) is incorporated by reference. Further, sampling must be conducted in accordance with the sampling procedures discussed in Section 4.0 of the QAPP (Utah DEQ, 2006).

Collection of Ambient Air Samples

Potential contamination of samples is a fundamental concern when sampling particulate phase mercury in air. Absolute adherence to clean sampling protocols is essential. This includes but is not limited to:

- All supplies that contact samples must be acid cleaned;
- All sample containers must be handled with particle-free gloved hands at all times;
- All sample containers must be bagged before and after sample collection; and
- Operators (i.e., individuals collecting the samples) must stand downwind of the sample during all sample installation and removal operations.

All sample collection procedures will be conducted outdoors (assuming that a Class 100 laboratory is not available at the collection site). In locales that experience extreme weather conditions, measures can be implemented, such as the use of small laminar-flow hoods in site trailers for loading and unloading sample filters from filter packs, to provide a relatively clean indoor environment appropriate for sample handling.

Following in Table 9, is a description of the method for collecting particulate phase mercury samples. The sampling equipment is described followed by specific procedures for sample collection. Note that references to a "gold trap" encompass traps made from gold-coated beads, gold-coated sand, or a solid gold matrix which has been demonstrated to produce equivalent results.

Sampling Equipment

Table 9. Equipment for Sampling Particulate Phase Mercury in Air

Equipment	Purpose
Pumps	Used for collection of mercury samples in ambient air. They must be intended for trace-level pollutant sampling. High efficiency oil-free, brushless pumps must be used and protected from the weather (i.e., well-sealed against rain, insulated and heated during the winter, and fan cooled during the summer.
Fiberglass Enclosure	Used as the sample box to house the filter packs and gold traps during sample collection.
Pole or Tower	For mounting the sample box. This must be long enough to mount the box at least 3 meters above ground level. Galvanized steel has performed well, providing sufficient strength in high winds. It may be wrapped with vinyl tape to prevent contamination from the metal surface.
Polyethylene Tubing with Quick Disconnect Couplings	Used to connect the vacuum lines from the pump to the sample box.
Dry Test Meter	Connected to the vacuum line between the pump and sample box for direct measurement of the volume of air sampled.
Flexible Latex Tubing with Quick Disconnect Couplings	Used inside the sample box to connect the vacuum lines to the filter packs.
Heat Tube	Used to prevent condensation in the gold traps, the heat tube is made from heat tape wrapped around a metal tube, insulated and covered with heat-shrink tubing and electrical tape. The heat tube is connected to a variable voltage transformer to provide constant low heat to warm the trap during sampling.

Teflon® Filter Pack	The 47-mm filter packs consist of three main components: a threaded inlet; a filter support base with ¼-inch tube ferrule nut, and a clampdown nut to secure the pack to the filter holder. An open inlet filter pack is used that protrudes several inches through the sample box during sampling. A plastic quick disconnect coupling is used for connection of the outlet to the vacuum line.
Rotameter	The meter is calibrated and used initially to set the flow rate for sampling. To prevent potential contamination, separate "flow check" filter packs are attached to the rotameters. The flow check filter packs have closed inlets with quick disconnect couplings on the inlet tubing for connection to the rotameter.
Field Test Data Sheet	Use to record all field activities

Collection of Particulate Phase Hg Samples

Because particulate phase mercury occurs at ultra-trace levels in the atmosphere and mercury has a high vapor pressure, the selection of sampling flow rate and duration must be given careful consideration. Typically, sample flow rates of 30 liters per minute for 12 to 24 hours are needed to collect enough particulate mercury for analysis.

Sample collection procedures have been subdivided into three parts: flow check procedures (Table 10), sample collection procedures (Table 11), and sample recovery procedures (Table 12). Each part is detailed below.

Flow Check Procedure

Table 10. Flow Check Procedures for Sampling Particulate Mercury in Air

Step	Procedure
1	Check the flow rate before each sampling event using a calibrated rotameter. To prevent potential contamination of the sample by the rotameter, a separate flow check filter pack is attached to the rotameter for this check. The flow check glass fiber filter must be replaced regularly but can be used several times.
2	Allow the sampling pump to warm up for at least 15 minutes prior to any flow measurement or adjustment.
3	The flow check filter pack is placed in the sampling box. The outlet is connected to the vacuum line and the inlet to the rotameter. After the system has stabilized, the flow rate is adjusted to the desired value (e.g., 30 Lpm) and is recorded on the FTDS. The flow check filter pack is then disconnected from the rotameter and vacuum line and removed from the sampling box.

Sample Collection Procedures

During all sample setup and removal operations, the operator must remain downwind of the sample to prevent contamination from shedding particles off of clothing, etc. Also, particle-free gloves must be worn when handling gold traps and prefilters.

Table 11. Procedures for Collecting Particulate Mercury Samples in Air

Step	Procedure
1	An acid cleaned sample filter pack with open cylinder inlet is loaded with a pre-baked glass fiber filter with the 'fibrous side' up, touching only the edge of the filter with a pair of acid cleaned Teflon® coated forceps. The filter pack is placed in the sample box.
2	To begin sampling, the vacuum line is connected to the outlet of the sample filter pack.
3	The following parameters are recorded on the FTDS: date, sampling location, time, ambient temperature, barometric pressure, relative humidity, dry gas meter (DGM) meter reading, rotameter reading, gold trap number, and DGM serial number.
4	Allow the sampler to operate for the desired time. At the end of the sampling period, record the parameters identified in Step 3 above on the FTDS. If the flow rates at the beginning and end of the sampling period differ by more than 10%, mark the filter cartridge as suspect.
5	Calculate and record the average sample rate for the filter cartridge using the following equation: $$Q_a = (Q_1 + Q_2) / N$$ where: Q_a = Average flow rate in Lpm Q_1, Q_2 = Flow measured at the beginning and end of sampling N = Number of points averaged.
6	Calculate and record the total volumetric flow rate for the gold trap cartridge using the following equation: $$V_m = (T \times Q_a)/1,000$$ where: V_m = total volume sampled at measured temperature and pressure (m^3) T = sampling time = $T_2 - T_1$ (minutes) T_2 = Stop time (minutes) T_1 = Start time (minutes)
7	Calculate the total volume at standard conditions (for example, 25ºC and 760 mmHg) using the following equation: $$V_s = V_m \times P_A/760 \times [298.15/(273.15+t_A)]$$ where: V_s = Total sample volume at standard conditions (m^3) P_A = Average barometric pressure during sampling (mmHg) t_A = Average ambient temperature during sampling (ºC)

Sample Recovery Procedure

Note that particle-free gloves must be worn during this procedure.

Table 12. Sample Recovery Procedures for Sampling Particulate Mercury in Air

Step	Procedure
1	The sample filter is removed from the filter pack and placed in an acid cleaned Petri dish using acid-cleaned Teflon®–coated forceps, touching only the edge of the filter.
2	The Petri dish is sealed with Teflon® tape and a label with a unique sample identification number is affixed to the cover of the Petri dish. The Petri dish is double bagged and returned to the laboratory for analysis.
3	The flow rate is checked after removal of the sample mercury traps using the flow check procedure outlined in Table 10. The flow rate and any deviations from the standard operating procedures during removal of the sample must be recorded on the FTDS.

Sample Storage

Particle-phase mercury filters can be stored in a freezer at -40 ºC until analyzed; however, holding times must not exceed twenty eight days from sampling.

B2.4 Mercury Speciation in Air

Mercury speciation can be determined using commercially available instruments available from Tekran. The company markets Model 1130 as an attachment to their Model 2537 Analyzer. The Model 1130 captures RGM and allows the Hg^0 to pass through. Using both units together, RGM and Hg^0 can be measured. The Model 1135 can also be included to monitor Hg_p. The cost of these units is high and training requirements are extensive. Recent research by groups in New Mexico (Caldwell, 2006), Nevada (Carr, 2006) and elsewhere has focused on the development of less expensive means of characterizing the dry deposition of atmospheric mercury species such as Hg_p, RGM, and Hg^0. In New Mexico, researchers used a manual sampling train with a KCl-coated denuder that mimicked the methodology employed in the automated Tekran 1130. The train measured airborne concentrations of Hg_p, RGM, and Hg^0. The results were then used in modeling the dry deposition of each species. This group also directly measured dry deposition of Hg_p and RGM using an ion exchange membrane as a passive surrogate surface (Caldwell 2006). In Nevada, researchers have used a variety of techniques, including the deployment of surrogate surfaces, to characterize dry deposition of atmospheric mercury species. Efforts are now underway to develop diffusive samplers that will measure RGM concentration and total mercury concentration. The results can then be used for calculations related to dry deposition. A future objective of the project is to perform comparative field tests between the diffusive samplers and the automated Tekran system utilizing the Model 1130 and 1135 speciation units. As these efforts mature, it is expected that the costs associated with characterizing dry deposition of Hg_p, RGM, and Hg^0 will come down. Given the location of these research projects and the potential for reduced costs, it is recommended that Utah DEQ remain cognizant of the progress made and, if and when appropriate, integrate the research results into its own efforts to characterize mercury deposition within the state.

B2.5 Surface Water (Total Mercury)

To evaluate waters for low level mercury using USEPA Method 1631, ultra-clean sampling methods are required. USEPA Method 1669 is commonly called a "Clean Hands/Dirty Hands" sampling method as it requires a two person sampling team with an additional person for recording information, labeling, etc. One person is designated "Clean Hands", the second is designated "Dirty Hands". All operations involving contact with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as "Clean Hands". "Dirty Hands" is responsible for preparation of the sampler (except the sample container itself), operation of any machinery, and for all other activities that do not involve direct contact with the sample.

Section 8 of Method 1669 is titled "Sample Collection, Filtration, and Handling". Four sampling procedures are described in detail in this section: directly into the sample container, using a grab sampling device, using a jar sampler, and a procedure for continuous-flow sampling using a submersible or peristaltic pump. Which procedure to use will be dependent on the logistics of the specific sampling site (stream vs. lake), depth of sample desired, etc.

Coordinated sampling may be possible at some of the locations involved in Utah's Division of Water Quality 2006-2007 Annual Monitoring Plan if they can provide information as a specific site is investigated.

To minimize contamination, sampling must be done as far as possible from roadways, bridges, and combustion sources, unless these sources are the sources being investigated.

Before samples are collected all sampling equipment and sample containers are cleaned in a laboratory or cleaning facility using detergent, mineral acids, and reagent water. After cleaning, sample containers are filled with weak acid solution, individually double-bagged, and shipped to the sampling site. All sampling equipment is bagged for storage or shipment. The laboratory or cleaning facility must prepare a large carboy or other appropriate clean container filled with reagent water for use with collection of field blanks during sampling activities. The reagent water filled container must be shipped to the field site and handled as all other samples containers and sampling equipment. At least one field blank must be processed per site, or one per every ten samples, whichever is more frequent.

Preservation with 5ml/L BrCl (0.2% per USEPA 1631e) must be done within 48 hours of sampling and may be done in the field or the laboratory. When preserved, the holding time for analysis is 90 days. Samples do not require refrigeration for shipping from the field to the laboratory.

B2.6 Surface Water (Dissolved Mercury)

Sampling for Dissolved mercury analysis follows the same procedures of USEPA Method 1669 with one required difference from the previously discussed surface water sampling. Samples for dissolved mercury analysis must be filtered through a 0.45µm capsule filter prior to preservation. This filtering may be done in a clean area set up in the field or may be done at the laboratory prior to preservation. It is imperative that filtering be documented when it is completed and by whom. A filtered blank must be prepared at the same time the samples are filtered.

B2.7 Geothermal Water (Total Mercury)

Water chemistry data for geothermal water within Yellowstone National Park show mercury levels ranging from 0-1520ng/L (McCleskey, Ball 2004). Geothermal springs and wells in Utah will be sampled following the same procedures of USEPA Method 1669 for "Clean Hands/Dirty Hands".

Many Hot Springs and Wells within the State of Utah are already involved in a program of water chemistry analysis. If any of these locations will provide information as a specific site is investigated, coordinated sampling may be possible.

B2.8 Snowpack Sampling

It is believed that snow particles "scrub" the air better than rain, and analysis of snowpack is a method for determining air deposition of mercury. No method for sampling the snowpack was found which complies with the "Clean Hands/Dirty Hands" philosophy of USEPA Method 1669 or USGS 2004, required for trace mercury analysis in waters, so the author has compiled the following with the understanding that the final melted sample be free from sampling contamination and be acceptable for analysis by USEPA Method 1631e:

- For each sample, sufficient snow must be sampled to get 0.5 Liters of water for preservation and analysis. Assuming a ten fold volume differential between the snow and water, approximately five liters of snow must be sampled, to err on the safe side, four one-gallon Ziploc bags are recommended.
- The sampling sites must be chosen to be free of avalanche danger.

- The sampling sites must be in areas of no disturbance, without canopy cover and protected from wind deposition and scouring—the intention is to sample the season's snowfall in as undisturbed a state as possible.
- Using a clean snow shovel, dig a pit the depth of the snow. Once this pit is complete, a two-person sampling team works in a "Clean Hands/Dirty Hands" mode. They will put on clean wind suits, and non-powdered gloves. "Dirty Hands" will clean the vertical phase of the pit using the pre-cleaned, rinsed, plastic snow shovel and "Clean Hands" will sample at four equidistant locations on the vertical plane filling a different clean gallon Ziploc bag at each of these four vertical locations (samples will be combined in a clean area after melting or in the laboratory prior to preservation). Samples need to melt, be combined and preserved with 5ml/L BrCl (0.2%) within 48 hours and the sample aliquot transferred to a 0.5 liter pre-cleaned glass bottle (the same as used with other water samples). The samples are then analyzed with the same procedure and QA/QC measures as surface water.

Record the snow depth, visible contamination, GPS location, personnel, weather observations, and description of snow stratification in the Field Notebook.

All equipment must be pre-cleaned, rinsed and wrapped in plastic prior to mobilization.

B2.9 Soil Sampling for Mercury

Soil samples will be collected to evaluate the mercury content in soils throughout the watershed. Measurements of total mercury in soil will be considered representative of the mass of total mercury transportable to surface water by means of physical erosion (i.e., mass wasting, runoff, and wind). Soil sampling locations will be selected to represent possible erosion pathways into surface water.

Soil samples will be collected according to the Standard Operating Procedures (SOPs) for Soil Sampling, detailed below. Following these SOPs will yield a sample suitable for mercury analysis by USEPA SW-846 Methods 7471 (for Total Mercury) and USEPA Method 245.5 (for Total Mercury-HgS). If samples are found to contain significant levels of mercury, further speciation studies may be done using USEPA Method 3200.

Specific details regarding soil sampling locations, depths, analyses, etc., will be provided in a site-specific Sampling and Analysis Plan (SAP).

Different geologic formations may have greater natural levels of mercury than other types of rock. When collecting soil samples, formation and soil characteristics of the sample will be noted. The following information must be recorded:

- Soil class (based on the Unified Soil Classification System [ASTM D-2487]);
- Moisture condition;
- Color (including mottling) using Munsell Soil Color Charts;
- Presence of organic material (e.g. rootlets, peat), nodules, shells, staining, etc., including estimated percentages;
- Size, grading, and angularity of clasts (including percentage of each); and
- Plasticity

Surface Soil

Surface soil samples will be collected in the following manner. When possible, soil samples will be collected from the area of least contamination to most contamination. Areas in close proximity to sample locations must remain undisturbed until all samples in the area have been collected.

Actual sample locations, depths, and other site specific details will be defined in the site-specific SAP.

Surface soil samples must be collected using a dedicated or decontaminated stainless steel spoon or trowel. Insert the sampling spoon or trowel to the appropriate sample depth and remove a sufficient volume of soil to fill the required sample containers.

As specified by the SAP, place the soil in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the sampling spoon or trowel. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove non-soil material (i.e. roots, stones, leaves, etc.) Then place the sample in the appropriate sample containers using the sampling spoon or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sampling location using a GPS.

Subsurface Soil

Subsurface soil samples can be collected using a hand auger, direct push technology, or hollow stem auger drill rigs. SOPs for sample collection using each of these methods are presented below

Hand Auger

Subsurface soil samples can be collected using a hand auger in the following manner: Rotate, drive, or push a decontaminated hand auger into the surface soil to the required depth. Slowly withdraw the auger from the soil. If a sample is not desired from that depth, remove the soil from the auger and repeat the first two steps.

Once the desired sampling depth is reached, replace the auger bucket with a pre-cleaned bucket or decontaminate the existing bucket. Use this clean auger bucket to collect the sample. Retrieve the auger and transfer the collected material into a Ziploc bag or stainless steel bowl, as required by the site-specific SAP. Repeat this process until sufficient sample volume has been retrieved.

Thoroughly homogenize the sample material using the sampling spoon or trowel. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove non-soil material (i.e. roots, stones, leaves, etc.) Then place the sample in the appropriate sample containers using the sampling spoon or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory.

Upon completion of sampling, backfill the borehole to grade using native soil, bentonite, or sand, as appropriate. Record the sampling location using a GPS.

Direct Push Technology

Subsurface soil samples can be collected using direct-push technology (DPT). DPT consists of a rugged, lightweight hydraulic drive point system mounted on a vehicle, trailer, or smaller hand held portable unit. The DPT system has the capability to drive sampling equipment for the collection of soil, soil gas, and groundwater samples. The DPT unit hydraulically advances hollow rods to a predetermined sampling depth. The media-specific (i.e. soil, vapor, water) type of DPT equipment is then employed.

The use of DPT may be a cost-effective alternative to conventional drilling methods for the collection of subsurface soils, soil gas, and groundwater samples. Prior to selecting DPT as a method for sample collection, consideration of the site geology, depth to groundwater, type of soils, site access, and topography must be made.

Prior to sampling, locate and mark potential sampling locations and ensure they are clear of utilities by following Utah's regulations for excavation (Call Before You Dig). In areas where utilities may be present, the first five feet of the boring will be advanced using a hand auger. Record the sampling location using a GPS.

Procedures for soil sampling using DPT technology are as follows. Generally, the operation of the DPT rig is conducted by a drilling subcontractor. DPT operations are detailed here for informational purposes.

<u>DPT Operation</u>

- Drilling equipment associated with sample collection (i.e. rods, tips, etc.) must be decontaminated before sampling and in between sampling locations.
- Assemble the sampling device by screwing the cutting shoe onto the bottom of the sampler (unless it is built-in). If using liners or sleeves, they will be placed in the sampler prior to assemblage.
- Thread the piston tip onto the piston rod.
- Thread the drive head onto the top end of the sampler.
- Slide the piston rod onto the sampler (the tip of the piston will slightly protrude from the cutting shoe) and reverse the piston stop pin onto the top of the piston rod.
- Thread the assembled sampler onto the lead push rod.
- Thread the drive cap onto the lead push rod and advance the sampler with the hydraulic hammer.
- Advance the sampler to the top of the predetermined sample interval by adding necessary push rods and then advancing them with the hydraulic hammer.
- Upon reaching the sample interval, remove the drive cap and lower extension rods down the inside of the push rods until encountering the top of the piston stop pin.
- Rotate the extension rods clockwise until the piston stop pin is removed from the sampler.
- Remove the extension rods with the attached piston stop pin.
- Replace the drive cap and advance the push rods and sampler through the sample interval.
- Remove the drive cap and replace it with the pull cap.
- Retract the push rods and sampler from the borehole (take care to secure the push rods remaining in the hole when removing a push rod to prevent them from falling back down the borehole.)
- Detach the sampler from the lead push rod upon retrieval.
- Disassemble the sampler, and remove the liner or sleeves.
- Repeat this process for sampling at additional depths.

Lithologic logging of soil at each boring location, including logging of sample soil, must be conducted. The following information will be recorded:

- Soil class (based on the Unified Soil Classification System [ASTM D-2487]);
- Moisture condition;
- Color (including mottling) using Munsell Soil Color Charts;
- Presence of organic material (e.g. rootlets, peat), nodules, shells, staining, etc., including estimated percentages;
- Size, grading, and angularity of clasts (including percentage of each); and
- Plasticity

<u>Sample collection procedures:</u>

Depending on the sampling containers to be used, several options exist for sample collection. If submitting a section of liner or sleeves containing the soil sample for analysis, seal both ends with

Teflon tape and plastic caps. Place samples in an ice-filled cooler pending transport to the analytical laboratory.

Otherwise, cut open acetate liner and remove a sufficient volume of soil to fill the required sample containers. Place the soil in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the dedicated or decontaminated stainless steel spoon or trowel. Then place the sample in the appropriate sample containers using the dedicated or decontaminated stainless steel spoon or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory.

Upon completion of sampling activities, backfill the boring with neat cement or bentonite grout, as required. Record the sampling location using a GPS.

Hollow Stem Auger

Subsurface soil samples can also be collected using a hollow stem auger drill rig equipped with a split-barrel (also known as split-spoon) sampler. Split-barrel samplers are routinely attached to the drilling rods of the drill rig. Split-barrel sampling is done when the sampler wants to obtain a relatively undisturbed discrete sample from a particular depth interval. This method provides best results in consolidated soils or competent material. Prior to selecting hollow stem auger split-barrel sampling as a method for sample collection, consideration of the site geology, depth to groundwater, type of soils, site access, and topography must be made.
Prior to sampling, locate and mark potential sampling locations and ensure they are clear of utilities by following Utah's regulations for excavation (Call Before You Dig). In areas where utilities may be present, the first five feet of the boring will be advanced using a hand auger. Record the sampling location using a GPS.

Procedures for hollow stem auger split-barrel soil sampling are as follows. Generally, the operation of the hollow stem auger rig is conducted by a drilling subcontractor. Drilling rig operations are detailed here for informational purposes.

<u>Drill Rig Operation</u>

- Drilling equipment associated with sample collection (i.e. drilling flights, rods, etc.) must be decontaminated before sampling and in between sampling locations. The split-barrel sampler must be contaminated prior to and after each use.
- Advance the borehole to the sampling depth specified in the SAP.
- Attach the sampler to the sampling rods and lower into the borehole. Do not allow the sampler to drop onto the soil or material to be sampled or penetrate the soil or material to any degree.
- Position the hammer and anvil above the sampling rods.
- Rest the dead weight of the sampler, sampling rod, and hammer and anvil assembly on the bottom of the borehole.
- Apply a series of seating blows.
- Mark the drill rods in successive 6-inch increments.
- Drive the sampler into the soil with hammer blows. Apply the hammer blows by using one of the following methods:
    - A trip, automatic, or semi-automatic hammer drop system which lifts the hammer and allows it to drop up to 30 inches unimpeded. If geotechnical samples are taken, allow the full 30-inch drop; or
    - A cathead to pull a rope attached to the hammer, provided the cathead is free of rust, oil, or grease and has a 6- to 10-inch diameter.
- Count the number of blows applied to each 6-inch drill rod increment until one of the following occurs:
    - A total of 50 blows are applied in a single 6-inch increment,
    - A total of 100 blows are applied,
    - Ten (10) successive blows result in no advancement of the sampler, or

    - The sampler is advanced the length of the split-barrel sampler (typically 18 or 24 inches).
- Record the number of blows in the field logbook or boring log.
- Retrieve and open the sampler.
- Repeat for each desired sampling depth.

Lithologic logging of soil at each boring location, including logging of sample soil, must be conducted. The following information will be recorded:
- Soil class (based on the Unified Soil Classification System [ASTM D-2487]);
- Moisture condition;
- Color (including mottling) using Munsell Soil Color Charts;
- Presence of organic material (e.g. rootlets, peat), nodules, shells, staining, etc., including estimated percentages;
- Size, grading, and angularity of clasts (including percentage of each); and
- Plasticity

<u>Sample collection procedures:</u>
Depending on the sampling containers to be used, several options exist for sample collection. If submitting a sleeve containing the soil sample for analysis, seal both ends with Teflon tape and plastic caps. Place samples in an ice-filled cooler pending transport to the analytical laboratory.

Otherwise, cut open acetate liner and remove a sufficient volume of soil to fill the required sample containers. Place the soil in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the dedicated or decontaminated stainless steel spoon, trowel, or spatula. Then place the sample in the appropriate sample containers using the dedicated or decontaminated stainless steel spoon or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory.

Upon completion of sampling activities, backfill the boring with neat cement or bentonite grout, as required. Record the sample location using a GPS.

B2.10 Sediment Sampling for Mercury

Sampling of sediments will allow for an understanding of transport and deposition of mercury in the water body. It will also allow for a better understanding of the methylation process and subsequent bioavailability of mercury.

Streams and rivers are dynamic bodies of water. The energy of a stream is directly related to the ability of the water to transport sediment. The greater the energy, the greater the distance of sediment transport. Typically the fastest velocity of a stream is near the center of the channel. Velocity is also greater on cut back versus point bars. Where stream velocity is slower, sediments have a tendency to fall out of suspension and accumulate. Sediment samples will be biased to these areas of higher sediment accumulation, such as along the sides of the stream on point bars and along insides of stream bends.

Sediments are mineral and organic materials situated beneath an aqueous layer. This aqueous layer can be flowing (e.g. rivers and streams) or static (lakes and ponds). The flow characteristics of a water body affect substrate particle size and organic matter content. Sediments containing fine particle size and high organic matter content are more likely to contain concentrated amounts of contaminants. Conversely, pollutants are unlikely to concentrate in coarse sediments with low organic matter contents. Therefore, selection of sampling locations can significantly affect analytical results and must be specified in the SAP.

For lakes, sediment samples will be collected near inlets, outlets, and near the deepest portions of the lake (if allowable). If sedimentation is significant, it may be possible to collect a shallow sediment sample as well as a deeper sediment sample. Comparing the samples will allow some indication as to whether depositional history has changed with time. Samples will be collected at times of low water levels. Low lake levels indicate of low influx of water, meaning the lake has lower turbulence and turnover. These conditions support sediment falling out of suspension.

Stream sediment samples will also be collected at times of low water flow. Sampling at low water levels will allow for sampling of more undisturbed sediments. For streams/rivers, sediment samples will be collected near the furthest upstream reaches and at intervals along the stream. The following SOPs for sediment sampling are based on USEPA Environmental Response Team SOP# 2016 for sediment sampling, and the Lake and Stream Bottom Sediment Sampling Manual prepared by the Resources Information Standards Committee of the Integrated Land Management Bureau, British Columbia.

If the presence of mercury is determined in sediment and there is a demonstrated need for depositional history, sediment dating, and/or methylation and demethylation rates, additional sediment core samples may be collected and analyzed to meet these needs.

Sediment Sampling Equipment

Sediment samples can be collected directly using a shovel, scoop, trowel or auger. Bottom sediments can also be collected using grab samplers or core samplers. Grab samplers can be collected indirectly using a remotely activated device such as a dredge. These grab samplers are easy to use and can obtain a large quantity of sediment for sampling. They are ideal for assessing more recent pollutant inputs.

Grab samplers commonly have a set of jaws that shut close when lowered to the sediment. The Ekman and Ponar dredges, discussed below, have vented or hinged tops that allow water to flow through the dredge as it is lowered to reduce sediment disturbance. This presents a disadvantage upon retrieval of the sampler, as fine sediment particles can be carried away by the flowing water. The Ekman dredge is best suited for soft, fine-grained sediments such as silts and clays. The Ponar dredge is best suited for fine to coarse grained material. Other types of grab sediment sampling dredges are also available commercially, such as the Peterson dredge. The Peterson dredge is best suited for collecting hard bottom materials such as sand, gravel, and firm clay. Its operation is similar to that of the Ekman and Ponar dredges, therefore, SOPs for use of the Peterson dredge are not provided. Other types of dredges are also available commercially.

Core samplers are used to collect a vertical profile of sediments and can provide information on the vertical distribution of contaminants and sediment deposition. Therefore, they are ideal for long-term or historical inputs. Core samplers consist of a tube that enters the sediment, either by free falling from a specified height (usually approximately 10 to 15 feet) or by direct pressure into the sediments. A valve at the top of the sampler closes and prohibits the sediment contents of the tube from falling out through the use of a vacuum seal. A common type of core sampler is the Kajak-Brinkhurst sampler, with variations on this design available commercially.

The distribution of streambed particle sizes can be determined using sediment traps, or samplers (McNeil sampler or Freeze core sampler) that collect an entire portion of the stream bed. Sediment traps are open buckets that are filled with clean gravel, placed in the streambed, and collected at a later date.

Prior to sampling, locate and mark potential sampling locations. Sediment sampling locations must be approached from downstream, so as to minimize cross contamination and turbidity issues. Actual sample locations, depths, and other site specific details will be defined in the site-specific sampling and analysis plan. Non-dedicated sampling equipment must be decontaminated between sampling locations.

Sediment Sampling in Shallow Rivers

Shallow Sediment Sampling with Trowel or Scoop
Surface sediment samples (0-0.5 feet below ground surface (bgs)) collected from beneath a shallow aqueous layer (0 to 2 feet bgs) can be collected using a dedicated or decontaminated stainless steel spoon or trowel, similar to the collection of surface soil samples. Insert the sampling spoon or trowel to the appropriate sample depth and remove a sufficient volume of sediment to fill the required sample containers.

Place the sediment in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the sampling spoon or trowel. The samples collected must be representative of the sediment and free of rocks and debris. If necessary, remove surface water and non-sediment material (i.e. roots, stones, leaves, etc.). Care must be taken to retain fine sediments. Then place the sample in the appropriate sample containers using the sampling spoon or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

Deep and rapidly flowing water make this sampling technique difficult and less accurate. Additional sediment sampling techniques are discussed below.

Shallow Sediment Sampling with Hand Auger from Beneath a Shallow Aqueous Layer
Surface sediment samples (0 to 0.5 feet bgs) collected from beneath a shallow aqueous layer (0 to 2 feet bgs) can be collected using a hand auger with sampling bucket or tube. The hand auger could be used to collect samples at greater depths; however, the difficulty of sample collection increases with depth.

Rotate, drive, or push a decontaminated hand auger at a 0 to 20 degree angle from vertical into the sediments to the required depth. This angle minimizes sample spillage upon extraction. Slowly withdraw the auger from the sediment and water.

Once the desired sampling depth is reached, collect the sample by retrieving the auger and transferring the collected material into a Ziploc bag or stainless steel bowl, as required by the site-specific SAP. Repeat this process until sufficient sample volume has been retrieved.

If specified by the SAP, thoroughly homogenize the sample material using the sampling device. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove surface water and non-sediment material (i.e. roots, stones, leaves, etc.). Care must be taken to retain fine sediments. Then place the sample in the appropriate sample containers using the sampling scoop or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

Deep Sediment Sampling with Hand Auger from Beneath a Shallow Aqueous Layer
Deep sediment samples (0.5 to 1.5 feet bgs) collected from beneath a shallow aqueous layer (0 to 2 feet bgs) can be collected using a hand auger with sampling bucket or tube. The hand auger could be used to collect samples at greater depths; however, the difficulty of sample collection increases with depth. Additionally, this method is limited by the depth of the aqueous layer and the integrity of the borehole.

Rotate, drive, or push a decontaminated hand auger into the sediments to the required depth. Slowly withdraw the auger from the sediment and water. If a sample is not desired from that depth, remove the sediment from the auger and repeat the first two steps. Sediment cuttings will be disposed of far enough from the sampling location to minimize cross contamination.

Once the desired sampling depth is reached, remove the bucket from the hand auger and attach the tube. Slowly lower the hand auger with tube down the borehole, taking care to avoid contact with the sides of the borehole, which could cross contaminate the sample. Gradually force the auger tube into sediment to the desired depth. Do not hammer the tube as this could cause the boring walls to collapse. Collect the sample by retrieving the auger and tube, again taking care to minimize contact with borehole walls. Transfer the collected material into a Ziploc bag or stainless steel bowl, as required by the site-specific SAP. Repeat this process until sufficient sample volume has been retrieved.

If specified by the SAP, thoroughly homogenize the sample material using the sampling device. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove surface water and non-sediment material (i.e. roots, stones, leaves, etc.). Care must be taken to retain fine sediments. Then place the sample in the appropriate sample containers using the sampling scoop or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

<u>Subsurface Sediment Sampling with Coring Device from Beneath a Shallow Aqueous Layer</u>
Subsurface sediment samples (0.5 to 1.5 feet bgs) collected from beneath a shallow aqueous layer (0 to 2 feet bgs) can be collected using a system that includes a tube sampler, acetate tube, eggshell check valve, nosecone, extensions, and "T" handle, or drivehead. This system could be used to collect samples at greater depths; however, the difficulty of sample collection increases with depth.

Non-dedicated sampling equipment and containers must be decontaminated by triple rinsing the equipment with deionized water and using non-detergent soap if needed.

Assemble the coring device by placing the acetate tube into the tube sampler. Insert the eggshell check valve into the lower end of the sampling tube with the convex surface positioned inside the acetate tube. Screw the nosecone onto the lower end of the sampling tube, securing the acetate tube and eggshell check valve. Attach the handle (and extension rods, if needed) onto the upper end of the sampling tube.

If the "T" handle is used, place downward pressure on the device until the desired sampling depth is attained. After reaching the sampling depth, rotate the sampler to shear off the core at the bottom. Slowly withdraw the sampler from the sediment, unscrew the nosecone, and remove the eggshell check valve. Remove the acetate core from the sampling tube. Remove any water through the top of the sampler. Care must be taken to retain fine sediments. If submitting the acetate sleeve containing the sediment sample for analysis, the acetate core may then be sealed with Teflon tape and plastic caps. Otherwise, cut open acetate liner and remove a sufficient volume of soil to fill the required sample containers. If necessary, continue to collect additional sediment until sufficient material has been obtained to fulfill analytical requirements.

Place the soil in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the dedicated or decontaminated stainless steel spoon, trowel, or spatula. Then place the sample in the appropriate sample containers using the dedicated or decontaminated stainless steel spoon, trowel, or spatula. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove surface water and non-sediment material (i.e. roots, stones, leaves, etc.). Care must be taken to retain fine sediments. Then place the sample in the appropriate sample containers using the sampling scoop or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

If the drive hammer is used, attach the drive head, and drive the sampler into the sediment to the desired sampling depth. Record the length of tube that penetrated the sediment, and the number of blows required to obtain this depth. Remove the driver hammer and fit the keyhole like opening on the flat side of the hammer onto the drive head. In this position, the hammer serves as a handle for the sampler. Rotate the sampler to shear off the core at the bottom. Lower the sampler handle/hammer until it just clears the two ear-like protrusions on the drive head, and rotate 90 degrees. Slowly withdraw the sampler from the sediment. If using the drive head, pull the hammer upwards and dislodge the sampler from the sediment. Remove the coring device from the water, unscrew the nosecone, and remove the eggshell check valve.

Remove the acetate core from the sampling tube. Remove any water through the top of the sampler. Care must be taken to retain fine sediments. If submitting the acetate sleeve containing the sediment sample for analysis, the acetate core may then be sealed with Teflon tape and plastic caps. Otherwise, cut open acetate liner and remove a sufficient volume of soil to fill the required sample containers. Place the soil in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the dedicated or decontaminated stainless steel spoon, trowel, or spatula. If necessary, continue to collect additional sediment until sufficient material has been obtained to fulfill analytical requirements.

Then place the sample in the appropriate sample containers using the decontaminated or dedicated stainless steel spoon, trowel, or spatula. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

Sediment Sampling in Deep Rivers

Generally, sediment samples in deep rivers are collected from bridges or by boat. Dredge sampling devices are commonly used for deep river sediment sampling, and may also be used in shallow rivers if necessary. Deep river sediment sampling is rarely accomplished using a core sampler, because these devices require minimal flow. Core samples may be accomplished by hand, as specified above, in shallow rivers. Samples will be collected beginning with downstream locations and working towards upstream locations.

Surface Sediment Sampling with an Ekman or Ponar Dredge from Beneath a Shallow or Deep Aqueous Layer

Surface sediment samples can be sampled indirectly using a remotely advocated device, such as a dredge, and a mechanism to deploy it. This is accomplished by lowering a sampling device, the dredge, to the surface of the sediment using a rope, cable, or extended handle such as a crane. The device is activated remotely, and entraps sediment in spring loaded or lever operated jaws.

Ekman Dredge

The Ekman dredge is lightweight, with spring activated jaws, and is used for the sampling of moderately consolidated, fine grained sediments (silt and sand). The 15 cm x 15 cm size is common, but it varies in size. SOPS for sediment sample collection using the Ekman dredge are presented below.

Non-dedicated sampling equipment and containers must be decontaminated by triple rinsing the equipment with deionized water and using non-detergent soap if needed. Attach a sturdy nylon rope or stainless steel cable through the hole on the top of the bracket, or bolt the extension handle to the bracket. It is good practice to attach the other end of the rope or cable to the boat or bridge. Attach springs to both sides of the jaws. Place the jaws in an open position by placing trip cables over the release studs. Ensure the hinged doors on the dredge top open freely. Lower the sampler to a point 4 to 6 inches above the sediment sampling location. Drop the sampler onto the sediment. Trigger the jaw release mechanism (either by lowering a messenger down the line, or pressing the button on the upper end of the extension handle). Raise the sampler slowly and remove any water through the top of the sampler. Care must be taken to retain fine sediments. Open the dredge jaws and transfer the sample into a Ziploc bag or stainless steel bowl, as required by the site-specific SAP. Repeat this process until sufficient sample volume has been retrieved. If the dredge jaws were not completely closed, the sample must be discarded and a new sample collected. Unwanted sample material will be stored in a bucket until the completion of sample collection activities; then it may be dumped.

If specified by the SAP, thoroughly homogenize the sample material using the sampling device. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove surface water and non-sediment material (i.e. roots, stones, leaves, etc.). Care must be taken to retain fine sediments. Then place the sample in the appropriate sample containers using the sampling scoop or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

Ponar Dredge

The Ponar dredge is heavy, with weighted, spring or lever activated jaws. It is used to collect consolidated fine to coarse sediment. SOPs for sediment sample collection using the Ponar dredge are presented below.

Attach a sturdy nylon rope or steel cable to the top of the dredge. It is good practice to attach the other end of the rope or cable to the boat or bridge. Place the jaws in the open position and set the trip bar so that the sampler remains open when lifted from the top. Place the spring loaded pin into the aligned holes in the trip bar, if applicable. Slowly lower the sampler to approximately 2 inches above the sediment sampling location. Drop the sampler to the sediment. Slack on the line will release the trip bar or spring loaded pin. Pull up sharply on the line to close the dredge. Raise the sampler slowly and remove any water through the top of the sampler. Care must be taken to retain fine sediments. Open the dredge jaws and transfer the sample into a Ziploc bag or stainless steel bowl, as required by the site-specific SAP. Repeat this process until sufficient sample volume has been retrieved. If the dredge jaws were not completely closed, the sample must be discarded and a new sample collected. Unwanted sample material will be stored in a bucket until the completion of sample collection activities; then it may be dumped.

If specified by the SAP, thoroughly homogenize the sample material using the sampling device. The samples collected must be representative of the soil and free of rocks and debris. If necessary, remove surface water and non-sediment material (i.e. roots, stones, leaves, etc.). Care must be taken to retain fine sediments. Then place the sample in the appropriate sample containers using the sampling scoop or trowel. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

Sediment Sampling in Lakes

Lake sediment samples are generally collected from a boat using a dredge grab sampler or a core sampler. The water depth at each sampling location must be determined prior to sampling. Water depth can be determined using a variety of equipment, from a weighted rope to an electronic sounder.

Sampling procedures using the Ekman and Ponar dredges are presented above. Core samplers may also be used when sampling from a boat. Core sampler SOPs are described below.

<u>Subsurface Sediment Sampling with Coring Device from Lakes</u>

Open the valve and set the trigger mechanism. Fasten a rope securely to the corer, and attach the other end to the boat. Lower the corer to slightly above the sediment and then allow the corer to penetrate the sediment with its own weight. Other types of corers can be used for different sediments, but the general rule is that disturbance upon impact of sediment must be minimized. Send the messenger down to release the trigger. Slowly retrieve the sampler. Close the bottom opening with a stopper before removing the corer from the water to prevent loss of the sample. Remove the liner from the sample corer. Repeat this procedure to collect additional sediment cores. Lithological information such as sediment deposition, thickness, color changes, etc must be recorded.

Remove the acetate core from the sampling tube. Remove any water through the top of the sampler. Care must be taken to retain fine sediments. If submitting the acetate sleeve containing the sediment sample for analysis, the acetate core may then be sealed with Teflon tape and plastic caps. Otherwise, cut open acetate liner and remove a sufficient volume of soil to fill the required sample containers. Place the soil in a Ziploc bag or stainless steel bowl and thoroughly homogenize the sample material using the dedicated or decontaminated stainless steel spoon, trowel, or spatula. If necessary, continue to collect additional sediment until sufficient material has been obtained to fulfill analytical requirements.

Then place the sample in the appropriate sample containers using the dedicated or decontaminated stainless steel spoon, trowel, or spatula. Seal the sample containers and affix labels. Place samples in an ice-filled cooler pending transport to the analytical laboratory. Record the sample location using a GPS.

Sample Preservation, Containers, Handling, and Storage

Soil and sediment samples will be collected in wide mouth glass containers with Teflon lined caps. The sample volume depends on specific analyses to be conducted and will be specified in the SAP. Solid (soil and sediment) mercury samples have a holding time of 28 days. Samples will be stored at 4 degrees Celsius pending analysis.

Details regarding sample locations, depths, collection (grab or composite), analyses, and other site specific details will be presented in the SAP. Grab samples collected from a specific depth will be transferred directly from the sampling device into a labeled sample container. Composite samples or multiple grabs will be homogenized in a Ziploc bag or stainless steel bowl, and transferred using a sampling spoon or equivalent into the appropriate labeled sample containers.

An Option for Locating Mercury "Hot Spots" – A Screening Tool

Hot Spots are localized areas with unacceptably high levels of mercury. Equipment exists to screen these areas at reduced expense and in less time compared to a complete, quality sampling plan. The RA 915 Lumex Mercury Vapor Analyzer is equipment which may be used in areas of suspected high concentrations like mine tailings, at fault lines, etc., or in areas of unknown concentrations searching for hot spots or their sources.

B2.11 Fish Tissue Collection

This subsection describes the Quality Assurance/Quality Control procedures associated with fish and invertebrate tissue (section B2.12) sample collection. As described within the "Mercury Source Protocol, the overall sampling strategy will be based upon existing information that identifies areas with known mercury contamination issues. Since areas that contain fish with accumulated mercury represent the initial information from which Utah DEQ will begin studies to delineate mercury contamination sources, fate and transport etc., the QA/QC of this effort is vitally important. It is important to emphasize that the fish and invertebrate tissue mercury results will help determine the next steps for sampling. Therefore, there needs to be extra caution towards the preservation of tissue mercury concentration integrity. This subsection introduces the various methods by which QA/QC procedures can be applied during the field activities for tissue sampling.

Quality assurance/quality control (QA/QC) procedures apply to every facet of the field collection, analysis and data interpretation phases of a project. This protocol provides details regarding the actual sampling procedures to follow during sample preparation, shipment, and analysis. Field QA/QC activities will involve the following:

1. Train field personnel on methods, protocol requirements, and roles and responsibilities;

2. Follow consistent methodologies for sampling all sites;

3. Introduction of field QA/QC samples such as blanks, duplicates or sample material for matrix spike/matrix spike duplicate analysis; and

4. Implementation of field team cross examination of written records, such as field notes, chain-of-custody records, and field labels.

The following sections describe the specific field procedures for fish and invertebrate tissue sample collection, and describe the appropriate QA/QC samples to collect, and methods for field record QA/QC review. The proposed methods described herein adhere to the Standard Operating Procedures provided within the State of Utah's Standard Operating Procedures for fish tissue sampling (SOU, 2006). This protocol provides additional detail describing specific QA/QC components to the SOU standard procedures. Three types of field QA/QC samples will be collected, they include sample duplicates, matrix spike/matrix spike duplicates, and field blanks. These types of samples and their relative frequency of collection are described below. A considerable amount of overlap occurs in these approaches for fish and invertebrate sample collection; therefore, only those distinct method elements are described where appropriate. For further information, please refer to *Standard Operating Procedure for Collection and Preparation of Fish Tissue Samples for Mercury Analysis*, State of Utah Department Of Environmental Quality, dated August 2, 2006 and the Colorado Department of Public Health and Environment's QAPP (CDPHE, 2005).

Commercially Available Sampling Apparatus for Fish Tissue Collection

Prior to mobilization for field activities, it is important to coordinate the efforts with any other agency that may conduct similar studies. It may be possible to coordinate the proposed fish and invertebrate sampling with other studies and thereby coordinate the sampling activities. Other agencies such as the United States Fish and Wildlife Service (USFWS) and Utah Department of Natural Resources (DNR) Division of Wildlife Resources have fish and invertebrate sampling equipment as well as trained professionals accustomed to the sampling procedures. Prior to the field effort, these agencies will be contacted in order to achieve sampling coordination.

Two principle types of fish collection equipment used and described within this QAPP, include gillnets and electroshocking gear. An additional alternative would be the use of a 'purse seine' in appropriate habitat types. Gillnets and seines are commercially available from a variety of sources. Gillnets can be custom designed to include several panels of differing mesh sizes in order to capture a variety of fish species and size classes. Seines can also be custom designed in order to obtain an appropriate overall size. Readily available sources for gillnets and seines include Memphis Net and Twine and Sterling Net and Twine Co.

It is recommended that a gillnet designed to contain upwards of 4 to 5 panels of small to large mesh sizes be used in an effort to capture representative samples of fish (based on size and species) from each sampling location. A gillnet can be deployed to expose only those panels needed while keeping the rest rolled up and out of a fish's path. Useful techniques for gillnet deployment are described by the United Nations Fisheries and Aquaculture Department.

The USFWS, Utah DNR, and local universities may be contacted for assistance with electroshocking efforts. In addition, private companies may be contacted to provide resources necessary for the sampling effort. Initial points of contact for these services include:

Utah Department of Natural Resources
Division of Wildlife
1594 West North Temple, Suite 2110
P.O. Box 146301
Salt Lake City, UT 84114-6480
(801) 538-4700

Utah Fish & Wildlife Management Assessment Office (USFWS)
1380 S 2350 W
Vernal, Utah 84078
(435) 789-0351

Aquatic Consultants employs fisheries biologists, and their services in the Southwest region of the United States.

Sampling Procedures for Fish Tissue Collection

There are typically three phases to any sampling procedure: preparation and mobilization, sampling and processing, and demobilization (i.e., decontamination of equipment and departure from the field). Preparation and mobilization steps for fish sampling will include:

- Identification of suitable study areas as defined by existing information obtained from the Utah DEQ mercury biomonitoring studies. Utah DEQ has two recently produced maps that show where mercury in fish tissue sampling has occurred and where there are areas of concern that would serve as the initial focus for the proposed sampling efforts.

- Development of a definitive field sampling plan that identifies the locations within flowing and static environments to be sampled, the methods for sampling and the time period to begin and end the effort (access agreements, if necessary, will be obtained prior and used as selection criteria for sampling locations);

- Determination of sample identifier scheme (sample numbers, location codes, among others) for each sample, location, and sampling event;

- Collection of field QA/QC samples at an appropriate interval;

- Completion of the appropriate scientific collection permit(s) from the appropriate agencies;

- Coordination of sampling efforts with appropriate agencies in order to expedite completion of sampling program objectives;

- Review and understanding of the methods presented in this QAPP and identification of roles and responsibilities of each team member;

- Acquisition, preparation, and maintenance of sampling equipment and gaining a complete understanding of proper uses;

- Acquisition and agreements with the appropriate analytical laboratory that will receive fish tissues for analysis by analytical methods, as defined within this QAPP, and;

- Health and safety training considerations such as location of nearest hospital, definition of ‘buddy system’ protocols and emergency communication methods (all to be defined by the field team for each field event).

Once the above items have been addressed, the field team is free to mobilize for the actual field event. The field team leader and members will have copies of this QAPP and other appropriate guidance documents for this work effort (e.g., work plan) on hand. Cellular phones or other intra-team communication devices will be kept on hand at all times. Field progress will be relayed to appropriate team personnel, including the analytical laboratory, to aid in an effective field sampling event.

The actual sampling of fish tissues will be accomplished with the use of gillnets and/or electroshocking methods. Both types of sampling can be used for flowing and static environments; however, electroshocking is more appropriate for flowing aquatic environments, and gillnets for static. The actual sample location and type of sampling method will be determined before mobilization in order to complete the effort in a timely manner.

Before sampling begins, it is important to recall the objectives of the sampling effort which are summarized as follows:

- Collection of representative fish species from the two target trophic levels, including game fish and bottom dwelling fish is recommended if available;
- Mercury accumulation generally increases with age and size (length or weight). As a result, it is important to collect larger sized fish for analysis;
- A minimum sample wet weight mass of 200 grams is needed for complete analysis with appropriate detection limits;
- Whole body adult fish samples will be taken from the bottom dweller species;
- The five largest fish for the dominant sport species will be obtained at each location;
- Contamination of tissue samples is a major possible source of error. Conscious effort towards keeping working surfaces and tools clean will be a paramount concern during the field effort;

During the preparation stage, the necessary field equipment is gathered, cleaned, and maintenance checks will be performed. The necessary equipment for the fish sampling effort will include (taken from Colorado, 2005):

- Fish collection gear (e.g. gillnets and/or electroshocking gear) and boat, if appropriate;
- Dip nets for collecting fish samples during electroshocking;
- Buckets, tubs and/or live wells made of plastic or aluminum for holding live fish for processing after capture;
- Weighing scales of sufficient capacity to weigh fish collected, with 1% full scale resolution;
- Measuring board calibrated in centimeters or inches;
- Stainless steel fillet knives and plastic kitchen cutting board;
- Large heavy-duty trash bags, Ziploc-brand or equivalent heavy duty freezer bags, Saran wrap or equivalent, and paper towels;
- Disposable non-powdered gloves vinyl or polyethylene gloves;

- Appropriate aquatic sampling clothing, including protective clothing for electroshocking aquatic habitats (e.g., hip boots, chest waders, neoprene gloves, among others);
- Field data forms or field notebooks, labels for samples, chain-of-custody forms and seals, indelible ink pens;
- Ice chests and ice.

Fish capture rates are often increased when sampling takes place in the evening or at night due to surface feeding patterns for fish species. The actual technique accomplished may require overnight sets for gillnets, or daytime electroshocking of flowing systems. This will be accomplished by recovering gillnets at first light after an overnight set. Boat electroshocking of static systems will take place at night along shorelines. Smaller aquatic systems will be sampled with a back-pack shocker, if necessary, during daylight hours due to safety concerns associated with navigation. Refer to Neilson, L and D.L. Johnson (1983) and others for more information pertaining to electroshocking and gillnet sampling strategies.

Once the fish are captured, they will be placed in live wells or buckets filled with water until they can either be filleted or frozen for transport to the laboratory. The samples will be processed to select the appropriate species and individuals to comprise investigative samples, and lengths and weights of fish will be recorded. Team members handling fish will wear gloves at all times while collecting and processing samples, and will minimize the amount of contact of each fish. Fish will be sorted into appropriate samples in the field and immediately placed on ice. Fillet samples from game fish can either be taken in the field or at the laboratory.

If fish fillets are prepared in the field, a working table will be set up and kept covered with plastic sheeting until the actual filleting takes place. Each fish is filleted, and the fillets are skinned and placed inside a Ziploc bag. Each sample is given a unique identifier that indicates the time, date, location, and type of sample collected. The sample identifier scheme will be pre-determined prior to the actual field mobilization. A label will be affixed to the sample Ziploc bag. The samples will be placed immediately on ice and frozen.

The bottom-dweller whole body fish samples will be handled as little as possible. All individuals that comprise the composite sample will be placed into the same Ziploc bag. The Ziploc bag will be labeled with the appropriate sample identifier information and immediately placed on ice.

Any surface or tool used for the sampling process will be cleaned between each sample process. For instance, if several fillet samples are being created, all knives and working surfaces will be thoroughly cleaned with on-site water, and rinsed with deionized water. The field member(s) responsible for sample processing will change their gloves between each sample and minimize contact with each sample. Sample processing time will be expedited because if prolonged, the process will lead to the loss of mercury from the fish specimens.

One field QA/QC sample will be collected for every ten investigative samples. The field QA/QC sample types include collection of duplicate samples; however, the laboratory may request collection of a random tissue sample for a matrix spike/matrix spike duplicate QA/QC analysis. Field blanks are not typically collected for tissue data, but it may be appropriate to collect a site surface water field blank if site water is routinely used to rinse the equipment. The following describes how QA/QC samples will be obtained for fish tissues by type (fillet vs. whole body) (CDPHE, 2005).

  - A ***fillet fish tissue duplicate*** will be created by retaining the right fillets of the composite species as the duplicate, and the left fillets as the investigative samples. A ‘right fillet’ is defined as the right side fillet portion when looking down upon a fish with the head pointed forward (belly down). It is important to create the duplicate from a composite sample that is able to provide sufficient mass for both the duplicate and the real sample in order to achieve minimum detection limit requirements (200 gm). A fillet tissue duplicate will either be collected at a 1:10 frequency, or 1 per sample event if the number of samples collected per event is less than 10.

- A ***whole body bottom dweller sample duplicate*** can be created by either sorting sufficient numbers of comparable bottom dwelling species from the same location to comprise two independent samples, or by submitting a robust composite sample (comprised of at least 400 g) to the laboratory for homogenization and splitting. The second method is preferred and likely to produce more valid duplicate results. This method, however, is better addressed through laboratory QA/QC protocols than field QA/QC protocols. A whole body tissue duplicate must either be collected at a 1:10 frequency, or 1 per sample event if the number of samples collected per event is less than 10.

- A ***matrix spike/matrix spike duplicate*** (MS/MSD) can be viewed as a 'triplicate' QA/QC sample where the investigative portion, a duplicate and a second duplicate are submitted from the same sample. For fish tissue, an MS/MSD will need to be collected from fillets and portioned into equal 'thirds'. A composited whole body will contain at least 600 g of mass and be separated into thirds in the field; or, similar to the duplicate approach, samples will be submitted to the laboratory for homogenization and division. An MS/MSD helps to measure the efficacy of a tissue extraction process and may not be required. The laboratory will be contacted in order to determine if this field QA/QC sample type must be collected. An MS/MSD is typically collected at a 1:10 frequency, or 1 per sample event if the number of samples collected per event is less than 10.

- A ***field blank of site surface water*** must be collected during each sampling event to test whether the water is contributing any mercury cross contamination. A site surface water field blank must either be collected at a 1:10 tissue sample frequency, or 1 per sample event if the number of tissue samples collected per event is less than 10. A field blank is created by simply collecting site water into a sample collection bottle (500 ml polyethylene). This sample may require special preservation (hydrochloric acid to a pH of 2) as per laboratory protocols.

Decontamination Procedures for Fish Tissue Sampling

Dirty field equipment is a possible source of mercury and other analyte cross contamination. All equipment (e.g., boat, waders, gillnets, fillet working table, knives, etc.) will be thoroughly cleaned with the use of non-detergent soap (e.g. Alconox) and a brush, triple rinsed in deionized water, and wrapped with plastic or aluminum foil (where appropriate) for transport to and from the field and between sampling locations. When in the field, the sampling team needs to be aware of all possible sources of contamination that can interfere with sample analysis results. For example, fish specimens dropped on the ground may not be appropriate for inclusion in sample processing. Fish tissue processing will occur in "open-air" environments away from vehicles, boat motors, gas cans, electroshocking generator, etc., which can all emit possible contamination. In addition, debris will be removed from the samples (twigs, dirt etc.) with as little disturbance to the sample as possible.

During the sample processing efforts at a specific sampling location, all equipment will be constantly rinsed with site water, followed by deionized water. Working surfaces and tools will be cleaned between each sample processing event. Team members handling fish will change gloves between each sample processing event and as needed to maintain a proper level of protection against potentially contaminated environmental samples. If field conditions are not conducive to specimen processing and/or could lead to conditions that increase the likelihood of mercury cross contamination, fish samples will be labeled, put in Ziploc bags, placed on ice, and processed (e.g., filleting) in a clean laboratory setting. The same field decontamination steps apply to a laboratory setting.

Fish Tissue Sample Preservation

Tissue samples are preserved by freezing, and samples are contained in order to prohibit contamination from external sources. Preservation is accomplished by completion of tissue preparation steps, as described above, to obtain the necessary tissue mass for sample analysis. The sample is placed into a Ziploc bag, all air is removed and the bag sealed, and then placed in another Ziploc bag and sealed in the same process. The inside bag will be labeled with either a laboratory-provided sample label or handwritten with the use of an indelible ink pen, or both. It is best to use a laboratory label and then secure with clear adhesive tape. As labels are completed and samples are packed, a field team-member will cross-check all information between the field notebooks, labels, and complete chain-of-custody forms.

The chain-of-custody and labeling information will be double-checked by a different field team member as a standard field QA/QC practice. The samples will then be chilled on ice in clean coolers and packed for shipping. Several samples may be placed in larger bags for shipping to contain any leaks. Once the samples are frozen solid, they are ready for shipment to the designated analytical laboratory.

A summary of steps that will be followed for sample preservation is as follows:

1. Place prepared sample into an new Ziploc bag, seal the bag removing all of the air;

2. Label bag with appropriate sample identifying information (sample number, location, date, time, sampler, method of sample collection, and analysis method, among others);

3. Place the labeled sample into a second Ziploc bag; seal the bag removing all air;

4. Place several samples within larger Ziploc bags or garbage bags to contain any leaks;

5. Cool samples until they are frozen solid, then prepare cooler(s) for shipment;

6. Complete the chain-of-custody forms as samples are packed for shipment using information gathered in the field and retained in field notebooks and transcribed onto sample labels. Check and cross-check the consistency of information between the sample labels, sample sheets, field sampling notebooks and the chain-of-custody forms.

Fish Tissue Sample Shipment

Samples will be shipped via overnight, guaranteed delivery methods. The cooler will be tracked during the shipment process with shipment receipts and tracking numbers supplied to the receiving laboratory. Chain-of-custody forms, with proper receipts retained, will be enclosed in a Ziploc bag and taped to the inside top of the shipping cooler. Samples will be pre-frozen and placed within enough ice to sustain $4^{o}C$ throughout the time of sample shipment. If it is not logistically possible to freeze the samples before shipment, they will be cooled as much as possible. Coolers will be sealed with two custody seal stickers, placed on opposing corners of the cooler and taped over and completely around the cooler with clear adhesive tape. Packing-fiber tape will be wrapped around the cooler to prevent opening during transit. Tape will be used to secure the cooler drain, cooler latch, and shipping labels.

The laboratory analyzing the samples will be contacted prior to shipment to ensure that the laboratory is prepared to receive, inventory, store, and analyze samples in a timely manner in order to meet necessary holding times requirements. Once the samples arrive at the laboratory, a representative will check the samples for holding time and temperature requirements. Any leakage or significant loss of temperature will be recorded as a source of potential error and noted in the laboratory analysis reports. The laboratory representative will then sign off on the COC as the current possessor of the cooler and its content, thereby completing the chain-of-custody for the samples. A copy of this final COC will be provided as part of the laboratory reporting package.

B2.12 Aquatic Macroinvertebrate Tissue Collection

Commercially Available Sampling Apparatus for Aquatic Macroinvertebrate Tissue Sampling

The collection equipment for sampling aquatic macroinvertebrates includes a D-frame kick-net and/or a dredge sampler. There are numerous alternative macroinvertebrate sampling equipment and methods that may be used for collecting representative samples in aquatic environments, depending on the type of sampling conditions. Specific information on the use of the samplers will be provided in a work plan, once site reconnaissance has been conducted. There are various suppliers for aquatic macroinvertebrate sampling equipment, including Forestry Suppliers and WildCo.

There are also equipment rental companies such as Geotechnical Services. Aquatic services companies can provide both equipment and trained personnel to aid in sample collection and processing. if it is determined that additional resources are required for the effort, beyond any established teaming efforts with appropriate agencies or institutions. Please reference USEPA's Rapid Bioassessment Protocol (RBP) for further information on collection of aquatic macroinvertebrates (Barbour, et. al., 1999).

Sampling Procedures for Invertebrate Tissue Collection

Similar to the fish sampling procedures, there are typically three phases to the invertebrate sampling efforts, including preparation and mobilization, sampling and processing, and demobilization (decontamination of equipment and departure from the field). Aspects of invertebrate sampling that are different from fish sampling include sampling objectives and the specific sampling equipment that is required. Depending on the diversity and complexity of habitats determined from initial site reconnaissance work, alternative sampling strategies may be employed, such as a more quantitative sampler designed for sampling in specific habitat conditions (e.g., fine-sediment substrate versus cobble/bolder substrates). The objectives of the invertebrate sampling effort are summarized as follows:

- A 100 meter (m) reach representative of local stream characteristics will be selected for sampling. Sampling will begin at the downstream end of the reach and proceeds upstream with a total of 20 jabs or kicks taken over the length of the reach. A single "jab" consists of forcefully thrusting the net into a productive habitat for a linear distance of 0.5 m. A "kick" is stationary sampling accomplished by positioning the net and disturbing the substrate for a distance of 0.5 m upstream of the net.

- Different types of habitat will be sampled in flowing environments in approximate proportion to their representation of surface area of the total macroinvertebrate habitat in the reach. The number of jabs taken in each habitat type will be recorded on the field data sheet. The jabs or kicks collected from the distinct habitats will be composited for later tissue analysis. One composite sample per habitat type will be collected.

Therefore, it will be noted that additional sampling beyond 20 kicks or jabs may be necessary to obtain the appropriate biomass. All deviations from sampling protocols will be noted in field logs.

- Sample will be transferred from the net to sample container(s). The wet weight will be measured to ensure that at least 200 grams of tissue sample have been obtained for analysis. The remainder of sample(s) will be stored for later enumeration and identification.

- It must be noted that flowing environments lend themselves to sampling strategies that may not be applicable to static systems if non-wadeable areas are selected for sampling (e.g., deep water areas in lakes and ponds). In the event that this occurs, alternative sampling equipment will be employed to collect aquatic macroinvertebrates samples, such as a Ponar or Eckman sediment sampler. This sampling equipment also provides a quantitative sample on an area specific basis.

- Composite samples of multiple invertebrate species occurring within a macrohabitat type will be collected. One sample per macrohabitat type (e.g., riffle, pool, or run) will be obtained, from areas where fish sampling occurred, as appropriate. The definition of invertebrate habitat types is provided within the sampling protocol.

- A minimum sample wet weight mass of 200 grams is needed for complete analysis with appropriate detection limits.

- One sample of each type of habitat per location will be obtained.

- Contamination of tissue samples is a major possible source of error. Conscious effort towards keeping working surfaces and tools cleans will be a paramount concern and taken into careful consideration throughout the field sampling effort.

The collection frequency for field QA/QC samples will be one QA/QC sample per ten investigative samples. Field QA/QC sample types will include duplicates and matrix spike/matrix spike duplicates, if requested by the laboratory analyzing the tissue samples. Field blanks are not typically collected for tissue data, but it may be appropriate to collect a site surface water field blank if site water is routinely used to rinse the equipment. The following describes how QA/QC samples will be obtained for invertebrate tissues.

  - An invertebrate composite ***duplicate sample*** will be created by either sorting sufficient numbers of invertebrate specimens from the same habitat type, from the same sample location to comprise two independent samples, or by submitting a robust composite sample (comprised of at least 400 g) to the laboratory for homogenization and splitting. The second method is preferred and likely to produce more valid duplicate results. This method, however, is better addressed through laboratory QA/QC protocols than field QA/QC protocols. A duplicate will either be collected at a 1:10 frequency, or 1 per sampling event if the number of samples collected per event is less than 10.

  - A ***matrix spike/matrix spike duplicate*** will be obtained by compositing sufficient invertebrate tissue mass from the same habitat type at a given location in order to obtain at least 600 gm of mass. This combined mass can either be separated into thirds in the field, or be submitted to the laboratory for homogenization and division. An MS/MSD helps to measure the efficacy of a tissue extraction process and may not be required. The laboratory will be contacted in order to determine if this field QA/QC sample will be collected. An MS/MSD is typically collected at a 1:10 frequency, or 1 per sampling event if the number of samples collected per event is less than 10.

- A ***field blank of site surface water*** may be collected during each sampling event to test whether the water is contributing any mercury cross contamination. A site surface water field blank will either be collected at a 1:10 tissue sample frequency, or 1 per sampling event if the number of tissue samples collected per event is less than 10. A field blank is created by simply collecting site water into a sample collection bottle (500 ml polyethylene). This sample may require special preservation (hydrochloric acid to a pH of 2) as per laboratory protocols.

The following equipment will be obtained for invertebrate tissue sampling:

- A D-frame or bottom sampling kick-net and/or sediment core, dredge, or grab sampler, (the type of sampling device will be dependent on site-specific conditions);
- Five gallon buckets, squeeze bottles and various sieve sizes for separating invertebrates from debris;
- Field tweezers and probe tools, white sorting trays, and magnifying glass, if necessary;
- Sample containers and exterior labels (internal labels also, if desired), and;
- Indelible pens.

Once a composite sample has been obtained, the sample will be transferred to a bucket with site water for initial processing. Large debris will be removed along with sediment, rocks, twigs, etc. It is important that the invertebrate tissue samples only contain specimens with no abiotic media, which introduces a potential source of mercury cross contamination. This may be most easily accomplished by sorting specimens from the remaining sediments and materials with the use of a white sorting tray and field tweezers. All invertebrates must be retained as part of the sample. The composite sample will be placed within a sample jar, labeled, bagged to contain any leakages, and then cooled on ice. All sampling equipment will be triple-rinsed with site water and deionized water before use at the next habitat sampling location.

Decontamination Procedures for Invertebrate Tissue Sampling

All sampling equipment used for collecting aquatic macroinvertebrates will be thoroughly pre-cleaned before each field mobilization effort. All field equipment will be rinsed with site water and deionized water between sampling locations. Field members responsible for sorting and processing aquatic macroinvertebrate samples will wear nitrile gloves and change between each discreet sample being processed.

Invertebrate Tissue Sample Preservation

The methods previously described within the fish tissue sample preservation subsection are appropriate for the invertebrate tissues.

Invertebrate Sample Shipment

The methods previously described within the fish tissue sample shipment subsection are appropriate for the invertebrate tissues.

B2.13 Moss Sampling QA Procedures

Quality assurance/quality control (QA/QC) procedures apply to every facet of the field collection, analysis, and data interpretation phases of a project. This protocol provides sampling procedures for collecting moss tissue and related data in the field, sample preparation and shipment, as well as for analysis of moss tissues samples. Field sampling QA/QC activities will involve: training field personnel on sampling methods, protocol requirements, and roles and responsibilities; consistently following standard methodologies for sampling activities at all sites; use of field QA/QC samples including trip blanks, field duplicates, and matrix spike/matrix spike duplicate analysis, and; implementing cross examination of written records, including field notes, chain-of-custody records, and field labels.

SAMPLING PROCEDURES FOR MOSS COLLECTION

The information provided for the moss tissue field sampling effort involves three phases, including preparation, sampling and processing, and equipment decontamination. However, it is important to note that field sampling efforts will also involve the development of a definitive field sampling plan that further identifies the specifics on sampling design, time and frequency of the study, and field sampling site locations. Sampling site locations will be based on desktop surveys and data searches, followed by field reconnaissance. Sampling sites will be located in areas where air deposition of mercury and mosses are known or suspected to occur. Results from initial sampling efforts will direct future sampling investigations.

Preparation for Field Sampling Efforts

A number of tasks must be addressed during the preparation phase of the field sampling effort, including:

- Team review and understanding of the methods presented in this QAPP and identification of roles and responsibilities of each team member;
- Determination of sample identifier scheme (sample numbers, location codes, date, among others) for each sample location and sampling event;
- Approved completion of the appropriate scientific biological collection permit(s) from the appropriate agencies;
- Acquisition, preparation, maintenance, and proper use of sampling equipment;
- Coordination of sampling efforts with appropriate agencies in order to expedite successful completion of sampling program objectives;
- Acquisition and agreements with the appropriate analytical laboratory that will receive moss tissues for analysis by analytical methods, as defined within this QAPP;
- Health and safety training considerations such as location of nearest hospital, definition of ”buddy system” protocols and emergency communication methods, all to be defined by the field team for each field event.

Additional items will be taken into consideration during the preparation phase of the sampling effort, including the sampling design and QA/QC considerations. The study design for properly determining potential combustion related deposition of mercury will be dependent on the estimated size of the depositional area(s), and will be directed by information obtained during field reconnaissance exercises. However, a *minimum* of eight moss samples, for a total of 15 grams (g), will be collected to characterize a sampling site. EPA SW-846 Method 7473 will be used to analyze moss samples, and the required biomass for this method is 5g. However, an additional 10g will be collected to provide for reanalysis of samples if necessary. Method 7473 will likely give sufficient sensitivity with smaller 0.2 g samples, however, the laboratory may need to demonstrate that the moss' homogeneity will allow sufficient precision at the smaller mass.

A biased sampling approach may be used if site-specific information is available, such as historic analytical mercury data for an area of concern, air-modeling results for suspected or identified combustion sources, or identified specific areas of concern. If site-specific information is not available, then a random sampling approach may be employed to identify areas of concern, such as a sample grid approach (e.g., "x" number of samples per "y" distance from suspected or known source).

One field QA/QC sample will be collected for every ten investigative samples. The field QA/QC samples will include collection of duplicate samples. The analytical laboratory may request collection of a random moss samples for a matrix spike/matrix spike duplicate (MS/MSD) QA/QC analysis. However, moss sample spikes may not be feasible, and recovery data for accuracy measurement may likely be obtained by analyzing plant SRMs for mercury instead. Field blanks are not typically collected for moss data, but it may be appropriate to collect an equipment blank or rinsate blank by pouring deionized water over decontaminated tools (e.g., hand trowel or scraper) and collecting the rinsate as a sample.

A sample duplicate will be collected by splitting a homogenized composite moss sample of sufficient mass (i.e., 30g) to attain minimum detection limit requirements for both the duplicate and investigative sample. Samples may be homogenized and split in the field, or submitted to the laboratory for processing. A duplicate moss sample will be collected for every ten investigative samples. One duplicate sample will be collected for a sampling event if the number of investigative samples collected is less than ten.

Should an MS/MSD pair be feasibly prepared, it can be viewed as a "triplicate" QA/QC sample where an investigative sample and two duplicates of that sample are submitted for analysis. Moss MS/MSD samples will be comprised from a homogenized 45g composite moss sample for a sampling site. Samples may be homogenized and split in the field, or submitted to the laboratory for processing. One MS/MSD sample may be collected for every ten investigative samples, or one MS/MSD sample will be collected per sampling if the number of investigative samples collected is less than ten.

A field equipment rinsate blank will be collected during each sample event to evaluate the effectiveness of equipment decontamination and storage practices. One equipment blank will be collected for every ten investigative samples, or one sample will be collected for a sampling event if the number of investigative samples collected is less than ten.

Sampling and Processing

Once preparation is complete, the field team will mobilize for the field event. Field sampling team members will maintain accessible copies of this QAPP and other appropriate guidance documents for this work effort (e.g., work plan) for each sampling event. Cellular phones or other communication devices will be used at all times. Field progress will be relayed to appropriate team personnel, including the analytical laboratory, to aid in an effective field sampling event.

The following equipment is necessary for moss sampling efforts (pre-cleaned where applicable):

- Hand trowel, flat scraper/putty knife, flat blade screwdriver, scissors, etc. for removed mosses, and a bucket and/or tray for sample collection and processing;

- Scale(s) with 1% full scale resolution and balance pan to measure the wet-weight of moss tissue sample;

- Ziploc heavy duty freezer bags or equivalent, plastic wrap, paper towels, and trash bags;

- Vinyl or polyethylene disposable, non-powdered gloves;

- Field data forms and/or field notebooks, labels for samples, chain-of-custody forms, custody seals, indelible ink pens;

- Ice chests and ice.

Sampling locations will be identified in the field using a global position system (GPS) and will be photographed as needed to adequately characterize the site, moss species, and sampling activities. All appropriate information (e.g., time and date of sampling, sampling location, weather conditions, surrounding land use/land cover, sampling activities being conducted, and a site map, among others) will be recorded.

Only epiphytic mosses not associated with soil will be sampled. This approach will reduce potential cross contamination of moss samples associated with mercury concentrations in soils unrelated to air deposition pathways (Mulgrew and Williams, 2000). Mosses will be identified in the field. Appropriate field guides resources will be used for moss identification, such as *Mosses: Utah and the West* (Flowers 1973), and efforts will be made to collect a single species common to the sampling areas. However, if this is not practical, then moss tissue samples may be composed of multiple moss species. Specific moss species will be targeted based on initial field reconnaissance work and information obtained from local and regional experts (e.g., State of Utah, U.S. Fish and Wildlife Service, universities and colleges, among others).

Moss samples will be collected from an appropriate height (e.g., at least 1.5 to 2.0 meters [m]) from at least two sides of a sampling substrate (e.g., tree trunk, rock face, or boulder), if possible. This approach will be used in order to minimize collecting moss tissue samples that may be associated with additional disturbances, such as exposure to mercury contaminated rain water or dust. A composite moss sample from one source (e.g., a rock or tree) will comprise one sample, with the composite comprised of a minimum of 8 moss samples from different rocks or trees, totaling at least 15g. The composite site sample will be comprised of green to greenish brown colored moss, which represents moss typically 3 to 5 years of age (International Cooperative Programme (ICP) 2005).

As best as can be managed, sample processing will occur in "open-air" environments away from possible contamination sources (e.g., roads and highways, urban areas, running vehicles). Foreign materials and detritus will be removed from the sample, including removal of all brown portions of the moss. Wet-weight measurements will be taken and recorded and species identification cross-checked by another field team member. A sample label will be firmly affixed to and/or placed in a new sample bag, along with the moss sample, the air removed and bag sealed, and then double bagged for added protection. The bag labels will be written with an indelible ink pen. As standard field QA/QC practice, field team members will cross-check information in the field notebooks and on the labels and chain-of-custody forms in order to ensure accuracy in the transcription process. The samples will then be frozen if possible, in a clean freezer. If the samples can not be frozen, then they will be packed on ice and cooled to $4^{o}C$ to slow decomposition. Several samples may be placed in larger bags to aid in sample management.

Once the samples are frozen, they will be packed and shipped to the laboratory for analysis. Chain-of-custody form receipts or copies will be enclosed within a Ziploc bag and taped to the top-inside of the cooler. Samples will be packed with enough ice to sustain $4^{o}C$ throughout shipment of the sample. Coolers will be sealed shut with two custody seal stickers at opposing corners, and taped over completely around the cooler with clear adhesive tape. Packing-fiber tape will be used secure the cooler lid so that it will not open during transit. Tape will be placed over the cooler drain, cooler latch, and shipment label. Samples will be shipped via overnight, guaranteed delivery methods. The cooler will be tracked during the shipment process with shipment receipts and tracking numbers supplied to the receiving laboratory.

The laboratory analyzing the samples will be contacted prior to shipment to ensure that the laboratory is prepared to receive, inventory, store, and analyze samples in a timely manner in order to meet necessary holding time requirements. Once the samples arrive at the laboratory, a representative will check the samples for holding time and temperature requirements. Any leakage or significant loss of temperature will be recorded as a source of potential error and noted in the laboratory analysis reports. The laboratory representative will then sign off on the COC as the current possessor of the cooler and its content, thereby completing the chain-of-custody for the samples. A copy of this final COC will be provided as part of the laboratory reporting package.

Decontamination

Contamination of moss samples is a major concern for this sampling effort. Field personnel will be aware of all possible sources of contamination that can interfere with sample analysis results and manage those sources in part through proper implementation of this QAPP. A focused conscious effort will be maintained during the field sampling efforts towards properly decontaminating, storing, and transporting sampling equipment, and preventing cross contamination of samples. Sampling equipment will either be decontaminated (e.g., hand trowels, scissors) or disposed (e.g., polyethylene gloves) before mobilizing to the next sampling location. Decontamination procedures for field sampling efforts will include washing field equipment with a stiff brush and non-detergent soap (e.g. Alconox), followed by a triple rinse with deionized water, and stored for clean transport.

B3. Sample Handling and Custody Procedures

Samples for laboratory analysis will be preserved as identified in Table A8 and held on ice. Field Sample Data and Chain-of-Custody Sheets will be completed and sent with the samples. Separate field data sheets will be maintained for each sampling event. Information recorded on data sheets will include: Project name, data and time of sampling events, water body name, basin name, numbers, general weather conditions, names of field staff, time of each sample or measurement, results and equipment ID numbers.

Samples will be transported on ice to the receiving laboratories via traceable, overnight transport.

B4. Analytical Methods

All parameters measured in the laboratory use these methods previously mentioned. USEPA 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (CVAFS) will be used for all water samples, including Surface Water, Dissolved Surface Water, Geothermal Water, Wet Air Deposition, and Snowpack.

Method 7473; Mercury in Solids and Solutions by Thermal Decomposition, amalgamation and Atomic Absorption Spectrophotometry will be used for fish, aquatic macroinvertebrates and moss.

Method 7471A; Mercury in Solid or semisolid waste (Manual Cold-Vapor Technique) will be used for determining Total Mercury in Soils or Sediments.

Method 245.5: Mercury in Sediment (Manual Cold-Vapor Technique) will be used for determining (Total Mercury - HgS)

Method 3200; Mercury species fractionation and quantification by microwave assisted extraction, selective solvent extraction and/or solid phase extraction will be used if mercury speciation of soils and sediments is desired.

B5. Quality Control

Quality Control Requirements are summarized in Table 13.

Duplicate quality assurance (QA) samples will be collected at a minimum of 10% of the total number of monitoring sites, or at least one duplicate per sample event.

Method Blank Analyses

A method blank is a "clean" sample (e.g. containing no analyte of concern), most often, Reverse Osmosis Deionized water, to which all reagents are added and analytical procedures performed. Method blanks are analyzed daily, or at a frequency of 1 per 10 samples, to assess possible contamination from the laboratory so that corrective actions may be taken, if necessary.

Quality Control Standards

Quality control standards traceable to NIST or USEPA are mid-range in the calibration curve and used to assess accuracy of instrument calibration. Control limits are based on historical data, or limits published by USEPA. Additional QC samples may be run at concentrations which represent the bulk of the samples tested and/or represents regulated levels.

Table13. Quality Control Requirements

Sample Media	Parameter	Method	LRB	LFB	SRM	DUP	LFM
			Proc. Blank	Laboratory Fortified Blank	Standard Reference Material	Duplicate	Matrix Spike
Wet Air Deposition	Hg (Total)	USEPA 1631e	±RL	±20%	NA	≤ 20%	±25%
Dry Air Deposition	RGM,Hg^0		±RL	NA	NA	≤ 40%	NA
Particulate in Air	Hg_p	7473	±RL	NA	NA	≤ 40%	NA
Surface Water	Hg (Total)	USEPA 1631e	±RL	±20%	NA	≤ 20%	±25%
Surface Water	Hg -- Dissolved	USEPA 1631e	±RL	±20%	NA	≤ 20%	±25%
Geothermal Water	Hg (Total)	USEPA 1631e	±RL	±20%	NA	≤ 20%	±25%
Snowpack	Hg (Total)	USEPA 1631e	±RL	±20%	NA	≤ 20%	±25%
Soil	Hg (Total)	7471, 245.5	±RL	±20%	±25%	≤ 40%	±40%
Sediment	Hg (Total)	7471, 245.5	±RL	±20%	±25%	≤ 40%	±40%
Fish Tissue	Hg (Total)	7473	±RL	±20%	≤ 40%	≤ 40%	±40%
Invertebrate Tissue	Hg (Total)	7473	±RL	±20%	≤ 40%	≤ 40%	±40%
Moss	Hg (Total)	7473	±RL	±20%	≤ 40%	≤ 40%	±40%

B6. Instrument/Equipment Testing, Inspection, and Maintenance

All field monitoring equipment will be tested for accuracy and /or calibrated in accordance with the manufacturer's instructions. In accordance with good laboratory practices, preventative maintenance programs, regular inspections, and careful operation of all instruments and equipment will be performed. Exact procedures are specific to Utah DEQ and are not included in this QAPP. They may be added later under guidance from Utah DEQ.

B7. Instrument Calibration and Frequency

All of the field and laboratory equipment and instruments used for this project will be calibrated at the frequency required by the method and according to the procedure and technical acceptance criteria set forth in the specified method, the laboratories' SOPs and per the manufacturers' recommendations.

All of the instrumentation that will used in the laboratory analyses will be calibrated prior to sample analysis and in accordance with the technical acceptance criteria of the analytical methods and the SOPs.

Balances are routinely calibrated or checked daily using NIST "Class S" weights, from 0.01 to 100.0 grams. Record the weights in a lab notebook located near the balance.

B8. Inspection/Acceptance of Supplies and Consumables

Field sampling staff will inspect and determine acceptability of supplies and consumables, before going into the field. Supplies and consumables will be obtained within a timeframe that allows re-procurement should something be found unacceptable.

B9. Non-direct Measurements

For purposes of this QAPP, we will consider non-direct measurements as mercury data already existing or collected under other studies. This ‘old’ or ‘outside’ mercury data may be value to this study and will be incorporated into completing this project if it meets the data quality objectives.

B10. Data Management

Data Management will be specific to Utah DEQ and is not addressed in this QAPP. It may be added at a later date pending guidance from Utah DEQ.

ASSESSMENTS AND OVERSIGHT

C1. Assessment and Response Actions

Assessment and Response Actions will be specific to Utah DEQ and are not addressed in this QAPP. They may be added at a later date pending guidance from Utah DEQ.

C2. Reports to Management

Reports to Management will be specific to Utah DEQ and are not addressed in this QAPP. They may be added at a later date.

DATA VALIDATION AND USABILITY

D1. Data Review, Verification and Validation

Data Review, Verification and Validation are project-specific and are not addressed here.

D2. Verification and Validation Methods

Data Review, Verification and Validation are project-specific and are not addressed here.

D3. Reconciliation with User Requirements

Reconciliation with User Requirements may be completed with guidance from Utah DEQ.

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LIST OF ACRONYMS

ASTM	American Society for Testing and Materials
BrCl	Bromine monochloride
COC	Chain-of-Custody
CVAFS	Cold-Vapor Atomic Fluorescence Spectrometry
DEQ	Department of Environmental Quality
DGM	Dry Gas Meter
DL	Detection Limit
DNR	Department of Natural Resources
DPT	Direct Push Technology
DQO	Data Quality Objectives
DSHW	Division of Solid and Hazardous Waste
DUP	Duplicate Sample
FTDS	Field Test Data Sheet
GPS	Global Positioning System
HCl	Hydrochloric Acid
Hg^0	Elemental Mercury
Hg_p	Mercury – Particulate Phase
HgS	Mercury Sulfide
HNO_3	Nitric Acid
KCl	Potassium Chloride
LASAR	Laboratory Analytical Storage and Retrieval Database
LCS	Laboratory Control Sample
LFB	Laboratory Fortified Blank (aka Laboratory Control Sample)
LFM	Laboratory Fortified Matrix (aka matrix spike)
LRB	Laboratory Rinse Blank (aka procedural blank)
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NADP	National Atmospheric Deposition Program
NFM	National Field Manual
$NH_20H•HCl$	Hydroxylamine Hydrochloride
$(NH_4)_2SO_4$	Ammonium Sulfate
NIST	National Institute of Standards and Technology
P.A.	p.a. Qualitat English: pro (or per) analysis
QA/QC	Quality Assurance/Quality Control
QAP	Quality Assurance Program
QAPP	Quality Assurance Project Plan
RBP	Rapid Bioassessment Protocol
RGM	Reactive Gaseous Mercury
RL	Reporting Limit
RPD	Relative Percent Difference
RSD	Relative Significant Difference
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SOU	State of Utah
SRM	Standard Reference Material
TGM	Total Gaseous Mercury
TWRI	Techniques of Water-Resources Investigations
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service